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Thermal Analysis of some propellants and explosives with DSC and TG/DTA

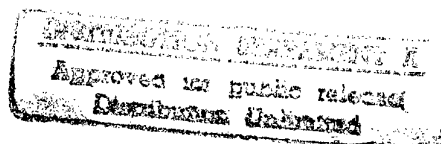
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Titel : Thermal Analysis of some propellants and explosives with DSC and TG/DTA
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STANAG 4515 geeft een beschrijving van de thermische analyse (TA) technieken (DSC - Differential Scanning Calorimetrie en TG/DTA - Thermogravimetry/Differential Thermische Analyse) waarmee de thermische karakteristieken van explosieve stoffen en kruiden bepaald kunnen worden.

Met behulp van DSC is het mogelijk de soortelijke warmte, onset-temperatuur en de enthalpie van zowel endotherme als exotherme reacties te bepalen. Met TG-metingen is het mogelijk gehaltebepalingen van componenten in explosieve stoffen en kruiden uit te voeren.

De TA-technieken kunnen ook gebruikt worden voor het bepalen van de verenigbaarheid van explosieve stoffen met andere materialen (onder andere verpakingsmaterialen). Dit is beschreven in STANAG 4147.

Tijdens dit onderzoek zijn thermogrammen van zestien explosieve stoffen en vijf kruiden opgenomen. Daarnaast is er met één kruid een verenigbaarheidsonderzoek uitgevoerd met vier polymeren.

TA blijkt een goede methode te zijn om explosieve stoffen en mengsels te karakteriseren (kwaliteitscontrole). De verkregen dataset zal als referentie gebruikt worden.

Ook blijkt dat TA een snelle indicatieve methode is voor het vaststellen van de verenigbaarheid van explosieven met andere materialen en kan als zodanig ondersteunend zijn voor de VST-resultaten.

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Symbols and definitions

DTA	Differential Thermal Analysis	
DSC	Differential Scanning Calorimetry	
TG	Thermogravimetry	
Tr	Temperature reference	°C
Ts	Temperature sample	°C
Tmin1	Max. peak temperature of first endothermic reaction	°C
Tmin2	Max. peak temperature of second endothermic reaction	°C
Tmelting	Melting point of the substance	°C
Tmax1	Max. peak temperature of first exothermic reaction	°C
Tmax2	Max. peak temperature of second exothermic reaction	°C
Tmass loss	Temperature of starting mass loss	°C
Mass loss1	Mass loss in first stage	%
To	Onset temperature exotherm reaction	°C
Teo	Extrapolated onset temperature	°C
DSC signal	microwatt	uW
DTA signal	microvolt	uV
TG signal	microgram	ug
Enthalpy (DSC)	microwatt/milligram	mJ/mg
Enthalpy (DTA)	microvolt.second/milligram	uv.s/mg

1 Introduction

At the request of DMKL, project A95KL486, an investigation was performed to create a database with characteristics of explosives and propellants. As a starting point, 16 explosives, 5 propellants and 4 polymers were investigated. All samples were characterized by the DSC and DTA/TG technique. From these measurements parameters such as heat capacity, mass loss as a function of temperature, onset temperatures, endothermic and exothermic enthalpies were determined.

These characteristic parameters will be used in the future as a reference for other explosives (mixtures) to improve quality control of new samples. They can also be used as an input for modelling other experiments such as heating tests with a large volume of explosive material (slow cook-off, fast cook-off, for example).

With DSC and TG, it is also possible to determine the compatibility of the explosives with different polymers, according to STANAG 4147. The results are related to the results of last year's test programme (Ref. 6).

2 Sample description

2.1 Explosives

The following explosives or explosive mixtures have been investigated using DTA/TG and DSC.

Table 1: Investigated explosives.

Sample number	Name of explosive	Chemical Name	Chemical Formula
E - 1	RDX (three samples)	Hexogen	$C_3H_6N_6O_6$
E - 2	HMX (two samples)	Octogen	$C_4H_8N_8O_8$
E - 3	Tetryl	Trinitro-2,4,6-phenylmethylnitramine	$C_7H_5N_5O_8$
E - 4	Pentrit	Pentaerythrol tetranitrate	$C_5H_8N_4O_{12}$
E - 5	TNT (two samples)	2,4,6-trinitrotoluene	$C_7H_5N_3O_6$
E - 6	TAGN	Triaminoguanidine Nitrate	$CH_9N_7O_3$
E - 7	DATB	Diaminotrinitro Benzene	$C_6H_5N_5O_6$
E - 8	Hexal	Mixture of RDX, aluminium, wax	—
E - 9	NTO	3-nitro-1,2,4-triazol-5-one	$C_2H_2N_4O_3$
E - 10	AP	Ammonium perchlorate	NH_4ClO_4
E - 11	Semtex	PBX based on PETN	—
E - 12	Composition B4	Mixture of RDX (60%) and TNT (40%)	—
E - 13	Sidewinder rocket propellant	Mixture of AP (83%), HTPB (10%) IDPI (3%) and others (4%).	—
E - 14	PBX - RU - 113	Mixture of RDX (78%), IDP (8%) and HTPB (14%)	—
E - 15	PBX - SNPE	Mixture of RDX (78%) and binder (22%), produced by SNPE (Fr)	—
E - 16	Black Powder	Mixture of potassium nitrate (75%), sulphur (10%) and charcoal (15%)	—

2.2 Propellants

The investigations are performed with one single base propellant, two double base propellants, one triple base propellant and a DEGN propellant.

The compositions of the investigated propellants are given in Table 2.

Table 2: Investigated propellants.

Propellant number	KB 5494	KB 6856	KB 6971	KB 6981	KB 7057
Nitrocellulose	20.5	96.2	80.0	59.4	62.1
Nitroglycerine	20.1		10.5	31.7	
Nitroguanidine	55.4				
DEGN					36.9
Ethylcentralite	3.7	1.9		0.94	0.24
Diphenylamine			1.0		
Dibutylphthalate			5.8		
Graphite		0.4	0.24	0.59	0.05
Moisture	0.20	1.3	0.67	0.42	0.43
Solvent		0.1	0.31		
Akardite II					0.45
Potassium salts		0.1	0.8	7.40	
Water				0.42	
Production year	1965	1987	1989	1990	1990

2.3 Polymers

For the investigation on the compatibility of propellants with polymers, the used polymers are described in Table 3.

Table 3: Used polymers.

Polymer	Chemical Name	Manufacturer
Nylon 6/6	Polyamid 6/6	Janssen Chimica
PMMA	Polymethyl methacrylate	Du Pont Lucite 2041
PVC	Polyvinylchloride	ACF
CA	Cellulose Acetate	BDH

2.4 Sample preparation

The explosive samples used were measured directly, while the propellants were ground before testing.

The sample pans used are made of aluminium, with an inner volume of 45 microlitres. The DTA/TG tests are performed with open cups, while the cups for the DSC tests were crimped to withstand an internal pressure of about 2 atmosphere.

Samples were representative of the material being studied and were prepared so as to have good thermal contact with the container.

3 Thermal analysis

Thermal Analysis (TA) is defined (Ref. 1) as: '*A group of techniques in which a property of the sample is monitored against time or temperature while the temperature of the sample, in a specified atmosphere, is programmed*'.

The programme may involve heating or cooling at a fixed rate of temperature change, or keeping the temperature constant, or any sequence of these.

The sample

The sample is often put into a pan which is then placed in contact with the sensor which is able to measure the particular property (e.g. melting, endothermic and exothermic reactions). A temperature sensor must also be present at (or very near) the sample to follow its temperature as the experiment progresses.

The sensor assembly then fits into a special furnace and the atmosphere around the sample is established. This is most important, because we may wish to study reactions with air or other reactive gases. The furnace is controlled by a temperature programmer.

If the sample is diluted with another substance, such as an inert reference material, then the composition of the mixture is given. This diluent can greatly alter the thermal conductivity of the sample. Even the particle size may alter the shape of the curve, especially where a surface reaction is involved.

The crucible

Sample holders are chosen because they do not interact with the sample during the course of the experiment. Different types of pans may well alter the heat transfer because of the different thermal conductivity. Therefore, the same type of pans were used in all the experiments.

For reactions with gases, special sample holders with free diffusion of reactant are available.

Heating rate

Experiments can be carried out at heating rates from 0 K/min (isothermal mode) through the normal heating rates of 10 K/min, to very high rates over 100 K/min, which are sometimes used to simulate the burning of materials (ignition of propellants or explosives). Since the rates of heat transfer are finite, the sample will react differently at different heating rates. In the experiments described in this report, 10 K/min is used.

Techniques

A single thermal analysis method does not always give sufficient information. For example, a downward peak produced by a DTA experiment means that an endothermic event occurs over this temperature range. It does not tell you whether this is a chemical reaction or a physical change, such as melting, or whether any gases are evolved. A TG experiment on the same sample may show a mass loss over this

temperature range, thereby ruling out melting, but still not identifying any volatiles. Conversely, if the TG curve showed no mass loss, the process of melting could be confirmed by direct observation of the sample. Combining several analytical methods gives a better profile of the changes taking place.

It is possible to carry out gas analyses by FTIR (Fourier Transform Infrared Analysis) or by MS (Mass Spectrometry).

Parameters

With the techniques used (DTA/TG and DSC) it is possible to measure many parameters, such as the onset temperature, extrapolated onset temperature, melting point, endothermic and exothermic enthalpies, mass loss as a function of temperature, glass transition, heat capacity, ignition temperature, purity etc..

In view of this programme, the onset temperature, extrapolated onset temperature, melting point, endothermic and exothermic enthalpies, mass loss as a function of temperature and heat capacity will be determined.

These tests are described in STANAG 4515.

The purpose of STANAG 4515 (Ref. 5) is to standardise test procedures for thermal characterisation of explosive materials using Differential Thermal Analysis (DTA), Differential Scanning Calorimetry (DSC) and Thermo Gravimetric Analysis (TGA). Information on the thermal characteristics of explosives is important from a safety and an operational point of view. Characteristics displayed on the thermograms can be used to monitor samples.

4 Test equipment

The equipment used in this research programme is the Seiko DTA/TG 320 instrument and the Seiko DSC-220-C, for measurements to low temperatures (-160°C) with an auto-cooling unit.

4.1 Differential Thermal Analysis/Thermogravimetry (DTA/TG)

Thermogravimetry (TG) is an old technique (Ref. 2) in which the mass of the sample is monitored against time or temperature while the temperature of the sample, in a specified atmosphere, is programmed.

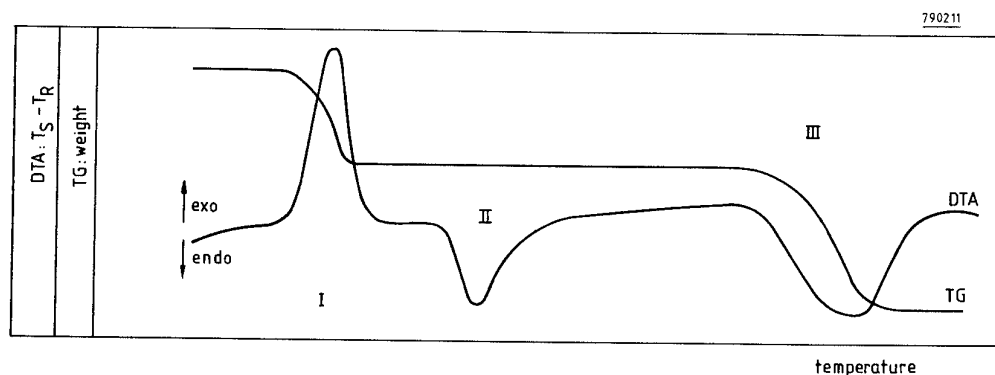


Figure 1: An example of a thermogravimetric curve and the differential thermal curve.
 T_S = sample temperature; T_R = reference temperature.

The weight is continuously monitored with a thermo-balance. The test equipment is capable of measuring changes of energy as well by Differential Thermal Analysis (DTA). With the combination of both techniques, temperature-dependent reactions like oxidation, reduction, dissociation and gas production (evaporation, loss of crystal water) can be investigated. A typical combination of a DTA and TG curve is shown in Figure 1.

Peak I is typical for an exothermal oxidation or reduction by which gaseous products are generated; peak II is a phase-transition which only results in a change in enthalpy, peak III can be describes the loss of crystal water.

The thermo-balance, Figure 2, consists of a DTA cell which is placed on a horizontal balance beam. The actual DTA cell is enclosed by an oven made of aluminium oxide. Temperatures from 30°C to 1300°C and heating rates from $0.01^{\circ}\text{C}/\text{min}$ to $100^{\circ}\text{C}/\text{min}$ can be applied.

The influence on the temperature from the surroundings is reduced by a metal reflector which surrounds the oven.

The balance room is protected against the penetration of corrosive gases from the oven by a small overpressure of a harmless gas, for instance dry air or nitrogen. The sample in the sample holder is exposed to the same gas. In this way it is possible to work under an arbitrarily chosen atmosphere. It is also possible to perform an experiment under vacuum.

The temperature of the sample can be measured with an accuracy of 0.5 K. For the weight loss, one must account with a resolution of $4 \cdot 10^{-9}$ kg.

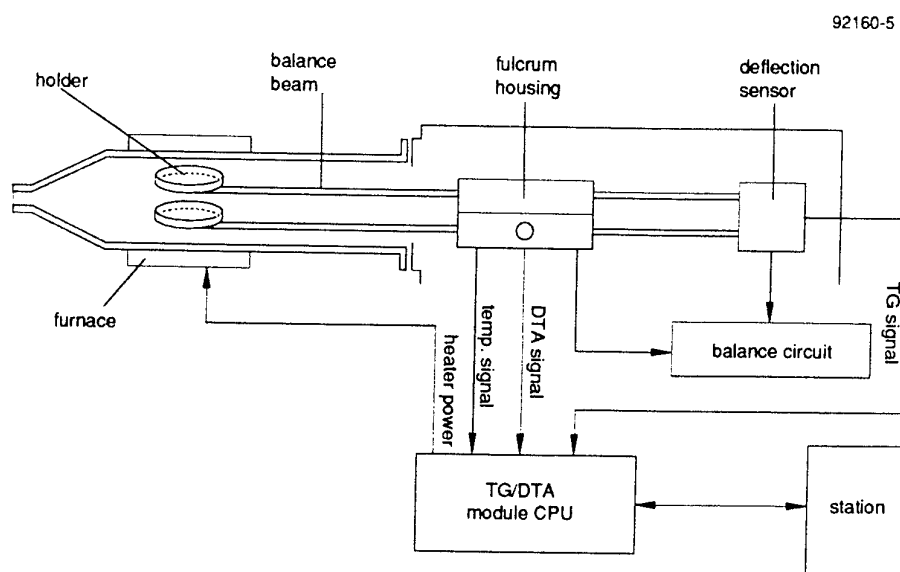


Figure 2: Seiko TG/DTA-320 thermo-balance.

4.2 Differential Scanning Calorimetry (DSC)

Heat Flux Differential Scanning Calorimetry (Heat Flux DSC) is a technique in which the heat flow (power) to a sample (pan) is measured as a function of the temperature whilst the sample is submitted to a certain temperature programme. During this temperature programme, the sample is either heated or cooled, which leads to endothermal or exothermal changes in enthalpy. These changes are caused by physical and/or chemical processes.

The Heat Flux DSC detects the difference in heat flow which is supplied to the sample and the reference material uniformly. The sample and the reference material are heated or cooled by a heat flow from the sink through the temperature

sensors. The heat sink has a much larger heat capacity than the sample and the reference. The heat flow difference between the sample and the reference material is measured. This difference is proportional to the temperature difference between the sample holder and the reference holder. This heat flow difference is quantitative, and is monitored as the y-axis signal in the DSC scan.

An example of a DSC curve is shown in Figure 3.

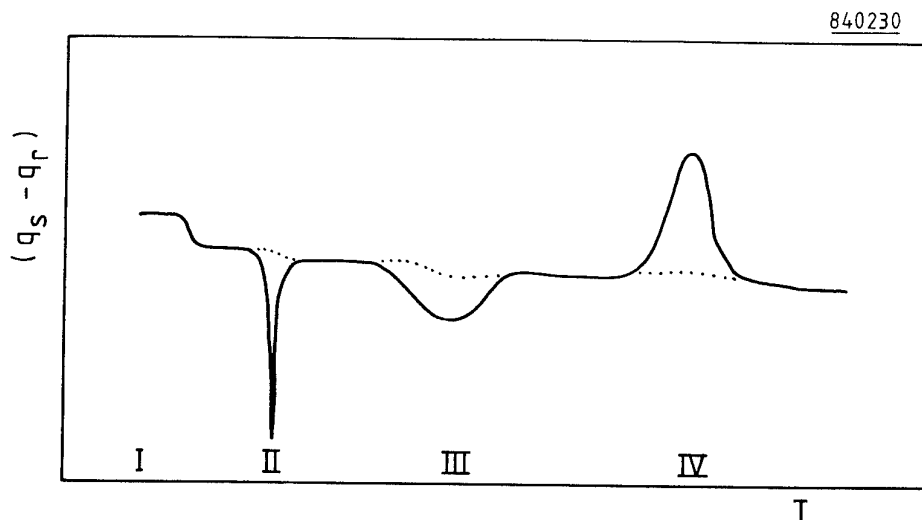


Figure 3: A typical DSC curve.

Four different types of reactions are shown in this figure:

- I : a baseline shift caused by a glass transition;
- II : an endothermal effect caused by melting;
- III : an endothermal effect caused by dissociation of the sample;
- IV : an exothermal effect caused by a chemical reaction.

The shape of the curve, the amount of energy involved and the temperature pattern observed are characteristic of a chemical substance, and, therefore, may lead to identification of the sample.

The onset temperature, T_o , is the temperature at which the substance or mixture shows the first effect of heat due to decomposition or reaction.

T_o depends on the sensitivity of the apparatus, on the sample mass and on the heating rate. When the experiment is used to establish the onset temperature (T_o) of the exotherm more accurately and/or is used to calculate kinetic data, a heating rate of 1 - 10 °C/min is appropriate.

A typical DSC curve of an exothermic peak is shown in Figure 4. The shape of the DSC curve is affected by several parameters such as heating rate, heat capacities and the heat conductivities of both the sample and reference material. The peak area is related to the total heat of reaction or decomposition.

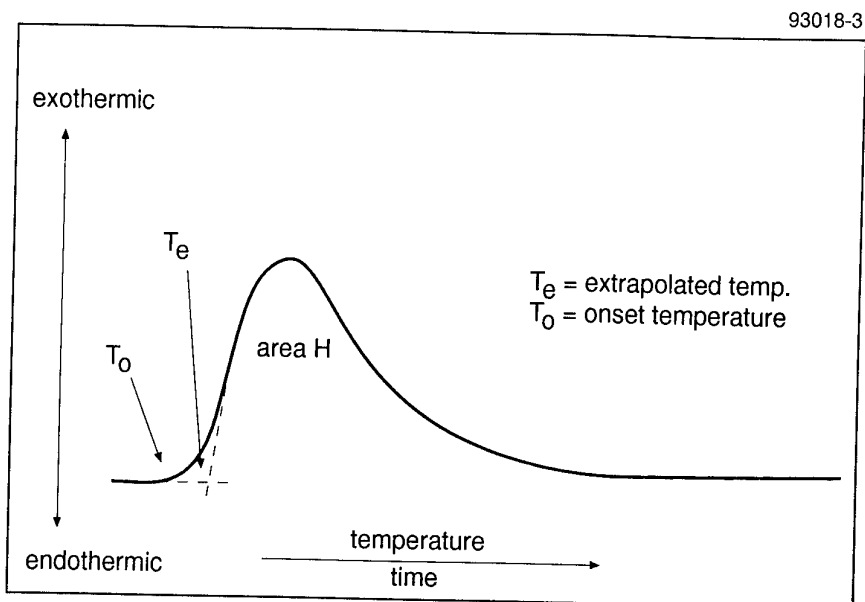


Figure 4: DSC curve of an exothermic reaction.

Various types of sample vessels can be used:

- aluminium containers with a volume of 15 microlitres and a maximum static overpressure of 30 atm.;
- silver containers with a volume of 15 microlitres and a maximum static overpressure of 50 atm.;
- stainless steel containers with a volume of 15 microlitres and a maximum static overpressure of 80 atm.;
- open or crimped aluminium containers with a volume up to 100 microlitres. The crimped containers have a maximum static overpressure of 2 atm.

The schematic design of the DSC apparatus (Seiko DSC 220C) is shown in Figure 5.

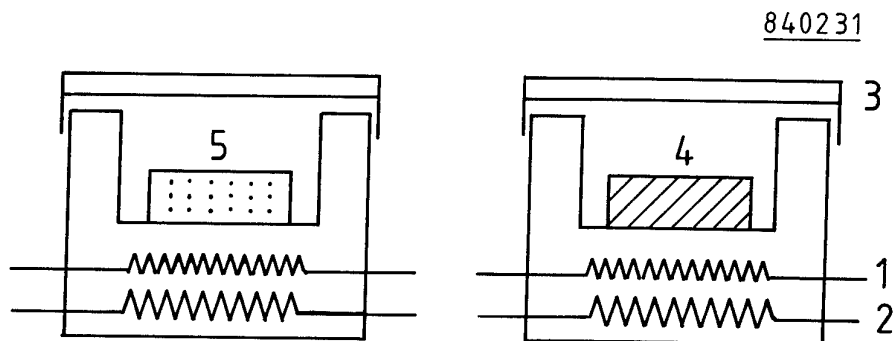


Figure 5: Schematic design of DSC.

- 1 heat sink;
- 2 sample;
- 3 reference;
- 4 sample holder;
- 5 temperature sensor;
- 6 furnace.

4.3 Calibration

For accurate work, it is essential to calibrate the temperature scale. For DSC instruments, the apparatus must also be calibrated for calorimetric sensitivity, which is also advisable for DTA. ICTAC (International Confederation for Thermal Analysis and Calorimetry) has approved a set of standard substances. Most commonly used calibration materials are indium, tin and zinc.

Each particular instrument may have calibration factors (e.g. slope control, temperature control etc.) already included in the computer software, but these must be checked from time to time. These factors depend on the heating rate, atmosphere and the used crucibles.

The TG equipment must also be calibrated to determine a weight calibration factor, by using certified mass.

5 Results

5.1 Calibration of the equipment

5.1.1 DTA/TG equipment

Since a DTA signal is used only semiquantitatively for measurements of endothermic and exothermic transitions, accurate calibration of the heat of transition/reaction is not generally necessary. However, if heat of transition/reaction is to be measured, calibration can be achieved by the measurement of heat of fusion of standard reference materials.

Because of the construction of the apparatus, temperature calibration to within $\pm 2^\circ\text{C}$ is considered acceptable. Generally, a two-point calibration will be performed, in this case with indium (melting point 156.6°C) and tin (melting point 231.9°C), because these points lie within the range of interest.

Calibrations are carried out using the same kind of crucible, heating rate, purge gas and flow rate as used for test measurements.

Table 4: Calibration constants for the DTA/TG equipment.

Description	Result	Factor
melting point indium	159.0	Toffset = -4.102 E+00 Tgain = 1.015 E+00 1dim.cal = -3.636 E-07
melting point tin	231.9	
DTA slope 30-300	-0.05 \Rightarrow -5.37 μV	TG span = 3.996 E+05
TG weight	20.020 mg	Factor = 1.129
Conversion factor	$\mu\text{V.s/mg} \Rightarrow \text{mJ/mg}$	

The resulting values are in agreement with the specifications of the apparatus.

5.1.2 DSC equipment

Calibration is performed by measurements of the heats of fusion and melting points of standard reference materials. The instrument is calibrated using the same type of sample crucible, heating rate, purge gas and flow rate as used for the test measurements. Generally, a two-point calibration will be performed, in this case with indium (melting point 156.6°C) and tin (melting point 231.9°C). Melting points onsets within $\pm 0.5^\circ\text{C}$ and heats of fusion within $\pm 2\%$ are generally considered to be acceptable.

Table 5: Calibration constants for the DSC equipment.

Description	Result	Factor
melting point indium	157.0	Toffset = 3.675 E-01 Tgain = 9.951 E-01 1dim.cal = -6.270 E-01
melting point tin	232.8	
DSC slope 30 - 300	289.3 \Rightarrow 454.2 μ W	DSC span = 1.655 E+05
melting indium	34.4 mJ/mg	
melting tin	67.3 mJ/mg	

These values are within the acceptance range of the equipment.

5.2 Thermal Analysis studies on explosives

5.2.1 Sample mass of investigated explosives

According to STANAG 4515 (Ref. 5), it is recommended to weigh the sample with a balance to at least 0.1 mg. The mass of the sample weights are given in Tables 6 and 7. For the DSC measurements, the weight of the sample cup including the sample is measured before and after the experiment to calculate the loss of weight.

Table 6: Sample mass of the explosives investigated by DTA/TG.

Sample number	Name of explosive	Mass of test sample [milligram]
E - 1	RDX (two samples)	3.226/3.907/3.902/4.312
E - 2	HMX (two samples)	4.683/4.405/3.081/4.568
E - 3	Tetryl	2.490/2.168
E - 4	Pentrit	2.990/3.547
E - 5	TNT	2.516/2.986
E - 6	TAGN	2.305/3.016
E - 7	DATB	2.760/3.584
E - 8	Hexal	3.827/2.769
E - 9	NTO	4.046/5.273
E - 10	AP	3.363/3.258
E - 11	Semtex	3.537/4.269
E - 12	Composition B4	5.074/5.257
E - 13	Sidewinder	3.126/3.688
E - 14	PBX - RU - 113	4.430/4.465
E - 15	PBX - SNPE	4.885/3.877
E - 16	Black Powder	3.583/3.431

Table 7: Sample mass of the explosives investigated by DSC.

Sample number	Name of explosive	Mass of test sample [milligram]
E - 1	RDX (two samples)	1.167/2.451/1.749/1.635
E - 2	HMX (two samples)	1.948/2.835/1.916/1.870
E - 3	Tetryl	1.327/2.054
E - 4	Pentrit	1.646/2.311
E - 5	TNT	1.310/1.862
E - 6	TAGN	1.553/2.122
E - 7	DATB	1.363/1.309
E - 8	Hexal	1.779/2.092
E - 9	NTO	3.015/2.519
E - 10	AP	2.030/1.852
E - 11	Semtex	3.881/1.684
E - 12	Composition B4	3.269/3.191
E - 13	Sidewinder	2.584/2.392
E - 14	PBX - RU - 113	3.110/2.941
E - 15	PBX - SNPE	2.992/3.081
E - 16	Black Powder	2.038/3.097

The test conditions are as follows:

DSC heating rate 10 °C/min
 purge gas nitrogen
 gas flow 50 ml/min

TG/DTA heating rate 10 °C/min
 purge gas nitrogen
 gas flow 50 ml/min

5.2.2 Thermograms of the investigated explosives.

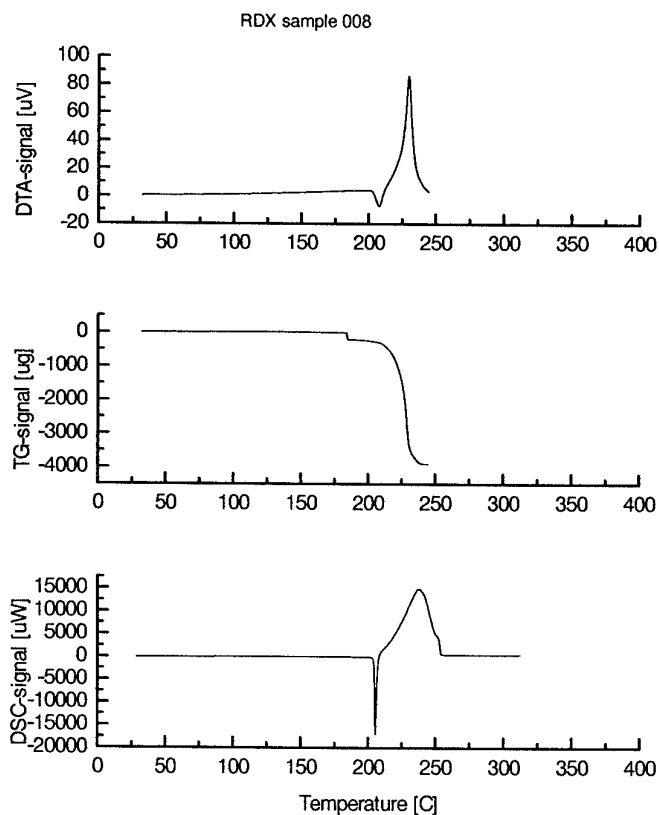
On the following pages, the results of the different explosives are presented in tabular form, followed by figures of the DSC and TG/DTA measurements.

See page 4 for an explanation of the abbreviations used.

RDX - Hexogen sample 94 EMK 008

DSC		DTA/TG		Effect
Tmin1	207/207	Tmin1	209/208	Melting transition peak maximum
Tmin2		Tmin2		Melting transition peak maximum
Tmax	237/239	Tmax	232/230	Decomposition exotherm peak max.
Tmelting 1	204/204	Tmelting 1	204/204	Onset of melting
Tmelting 2		Tmelting 2		Onset of melting
Tonset	209/209	Tonset	213/213	Decomposition exotherm onset
Te onset	211/215	Te onset	223/223	Extrapolated onset
		Tmass loss 1	178/178	Start decomposition
		Mass loss 1	100/100	Mass loss 1
		Tmass loss 2		Start decomposition
		Mass loss 2		Mass loss 2
		Total mass loss		
Endoth. Enthalpy 1	133/128	Endoth. Enthalpy 1	88/85	Melting Enthalpy
Endoth. Enthalpy 2		Endoth. Enthalpy 2		Melting Enthalpy
Exoth. Enthalpy	1189/1076	Exoth. Enthalpy	1212/1163	Sublimation Enthalpy, by DTA

For the interpretation see also sample 011, two pages further.



In both the DSC curve and the DTA curve, the peak downwards is due to melting of the RDX (endothermic reaction).

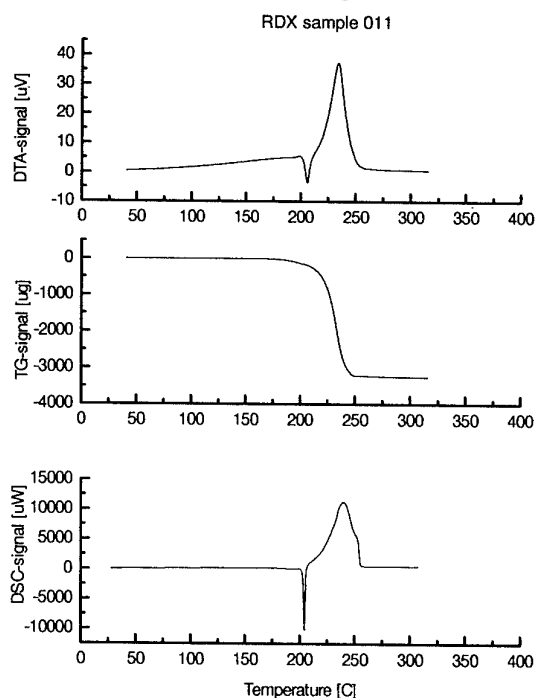
RDX - Hexogen sample 94 EMK 011

DSC		DTA/TG		Effect
Tmin1	205/205	Tmin1	207/208	Melting transition peak maximum
Tmin2		Tmin2		Melting transition peak maximum
Tmax	240/242	Tmax	235/232	Decomposition exotherm peak max.
Tmelting 1	203/203	Tmelting 1	203/203	Onset of melting
Tmelting 2		Tmelting 2		Onset of melting
Tonset	207/208	Tonset	211/212	Decomposition exotherm onset
Te onset	214/212	Te onset	221/222	Extrapolated onset
		Tmass loss 1	178/178	Start decomposition
		Mass loss 1	100/100	Mass loss 1
		Tmass loss 2		Start decomposition
		Mass loss 2		Mass loss 2
		Total mass loss		
Endoth. Enthalpy 1	112/100	Endoth. Enthalpy 1	72/68	Melting enthalpy
Endoth. Enthalpy 2		Endoth. Enthalpy 2		Melting enthalpy
Exoth. Enthalpy	1326/1302	Exoth. Enthalpy	1019/1215	Sublimation enthalpy, by DTA

Until RDX starts to melt at 203 °C, the DTA curve shows no transformation on heating. The melting point is in agreement with the value in Ref. 7, for DTA measurements.

The differences in endothermic enthalpies, between DSC and DTA, are due to the fact that molten RDX already starts to decompose before the melting process is over. DSC measurements are carried out with a slightly closed cup, so evaporation of the volatiles is limited (only in the beginning).

The endothermic enthalpy of sample 011 is about 80% lower, in contrast to sample 008. This could be explained by the purity of the sample. Sample 008 contains about 0.3% HMX whereas sample 011 contains approximately 21% HMX.

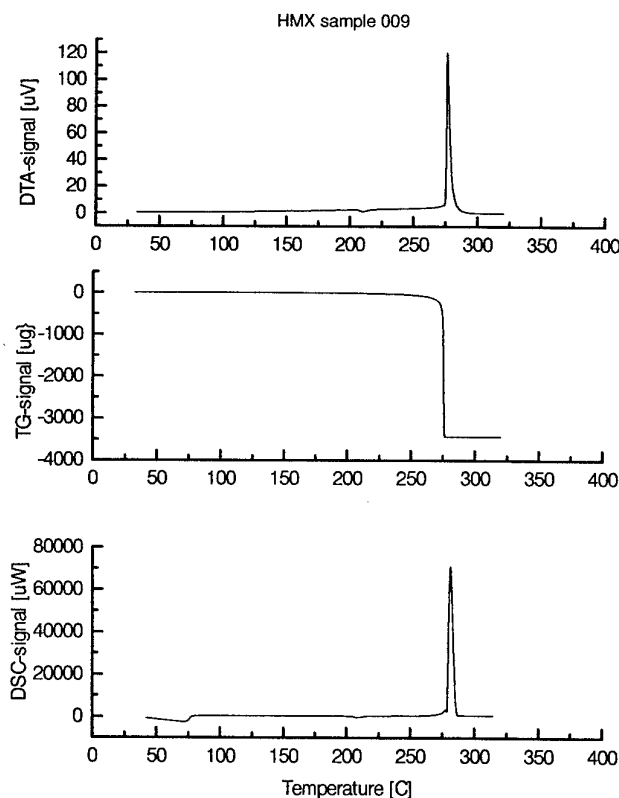


HMX - Octogen sample 94 EMK 009

DSC		DTA/TG		Effect
Tmin1	208/209	Tmin1	276/279	Melting transition peak maximum
Tmin2		Tmin2		Melting transition peak maximum
Tmax	282/282	Tmax		Decomposition exotherm peak max.
Tmelting 1	203/203	Tmelting 1		Onset of melting
Tmelting 2		Tmelting 2		Onset of melting
Tonset	242/242	Tonset		Decomposition exotherm onset
Te onset	265/265	Te onset	267/267	Extrapolated onset
		Tmass loss 1	253/253	Start decomposition
		Mass loss 1	75/75	Mass loss 1
		Tmass loss 2		Start decomposition
		Mass loss 2		Mass loss 2
		Total mass loss		
Endoth. Enthalpy 1	18/19	Endoth. Enthalpy 1		Melting enthalpy
Endoth. Enthalpy 2		Endoth. Enthalpy 2		Melting enthalpy
Exoth. Enthalpy	920/980	Exoth. Enthalpy	442/772	Sublimation enthalpy, by DTA

The DSC curve of HMX shows a strong exothermic peak at 282 °C. This peak is instantaneous after the melting of HMX, and is due to the self decomposition process.

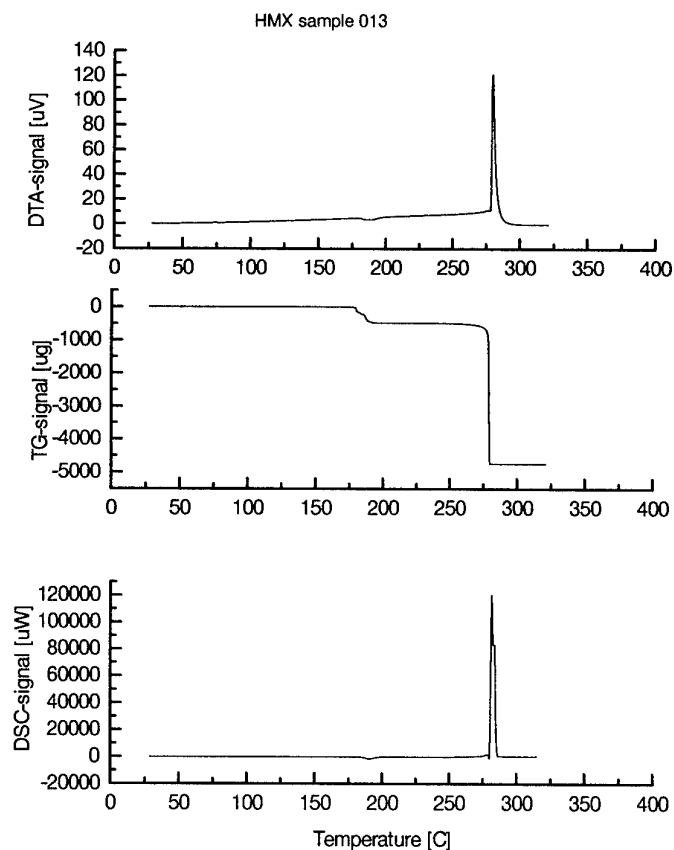
Specifications about the purity are not available for this sample. Based on the TG curve and the endothermic enthalpy, it may be concluded, that the purity is not 100%. When HMX is pure, the decomposition is normally nearly 100%. The big difference in the measured enthalpy by DTA is caused by evaporation of the HMX, when it starts to decompose.



HMX - Octogen sample 94 EMK 013

DSC		DTA/TG		Effect
Tmin1	-192	Tmin1		Melting transition peak maximum
Tmin2		Tmin2		Melting transition peak maximum
Tmax	282/281	Tmax	280/279	Decomposition exotherm peak max.
Tmelting 1	184/185	Tmelting 1		Onset of melting
Tmelting 2		Tmelting 2		Onset of melting
Tonset	242/240	Tonset		Decomposition exotherm onset
Te onset	268/266	Te onset	268/267	Extrapolated onset
		Tmass loss 1	178/179	Start decomposition
		Mass loss 1	10/10	Mass loss 1
		Tmass loss 2	262/264	Start decomposition
		Mass loss 2	90/90	Mass loss 2
		Total mass loss	100/100	
Endoth. Enthalpy 1	35/34	Endoth. Enthalpy 1		Melting enthalpy
Endoth. Enthalpy 2		Endoth. Enthalpy 2		Melting enthalpy
Exoth. Enthalpy	1125/700	Exoth. Enthalpy	384/390	Sublimation enthalpy, by DTA

Sample 013 shows a melting peak at a temperature of 184 °C, which compares with the beta-transformation in HMX. The purity of the sample is nearly 100% beta-HMX, according to the specifications of the manufacturer. The decomposition temperature of HMX is the same for both samples (013 and 009).

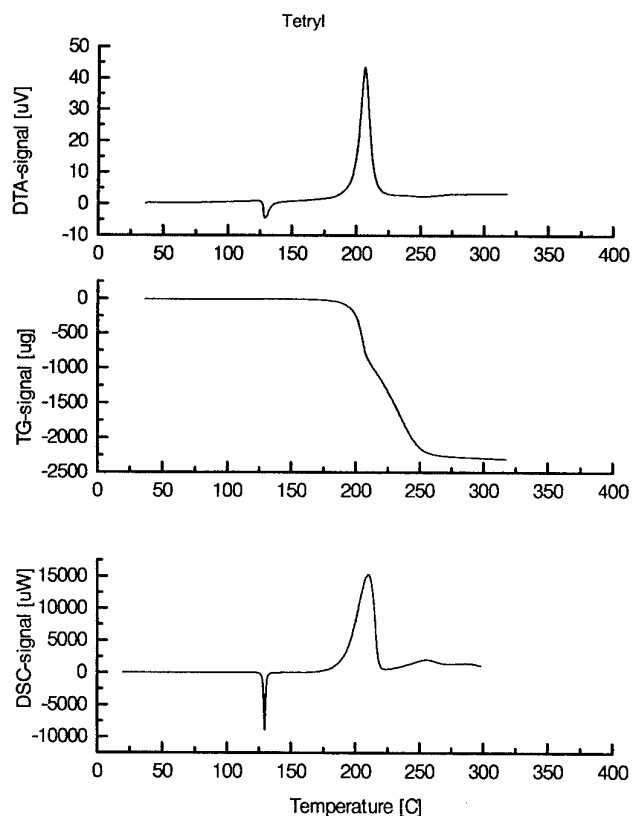


Tetryl - sample 94 EMK 019

DSC		DTA/TG		Effect
Tmin1	131/131	Tmin1	135/135	Melting transition peak maximum
Tmin2		Tmin2		Melting transition peak maximum
Tmax	208/208	Tmax	210/213	Decomposition exotherm peak max.
Tmelting 1	128/128	Tmelting 1	131/131	Onset of melting
Tmelting 2		Tmelting 2		Onset of melting
Tonset	167/167	Tonset	180/179	Decomposition exotherm onset
Te onset	192/194	Te onset	200/205	Extrapolated onset
		Tmass loss 1	178/179	Start decomposition
		Mass loss 1	37/38	Mass loss 1
		Tmass loss 2	212/216	Start decomposition
		Mass loss 2	55/54	Mass loss 2
		Total mass loss	93/92	
Endoth. Enthalpy 1	89/88	Endoth. Enthalpy 1	76/78	Melting enthalpy
Endoth. Enthalpy 2		Endoth. Enthalpy 2		Melting enthalpy
Exoth. Enthalpy	1141/1112	Exoth. Enthalpy	1144/1059	Sublimation enthalpy, by DTA

The sharp endothermic peak in DSC and DTA is due to the melting of tetryl. The TG curves show a two-step reaction, with a change in the decomposition rate at 212 °C. The maximum peak temperature (Tmax) corresponds with the value in Ref. 10 (211 °C).

The results are in agreement with the DTA and TG values given in Ref. 7. In Ref. 7, the mass loss measured by TG is about 77% in the temperature range to 225 °C, with a decreasing curve. So the mass loss of 92% is quite normal after a temperature of 300 °C.



OPentrit - sample 94 EMK 017

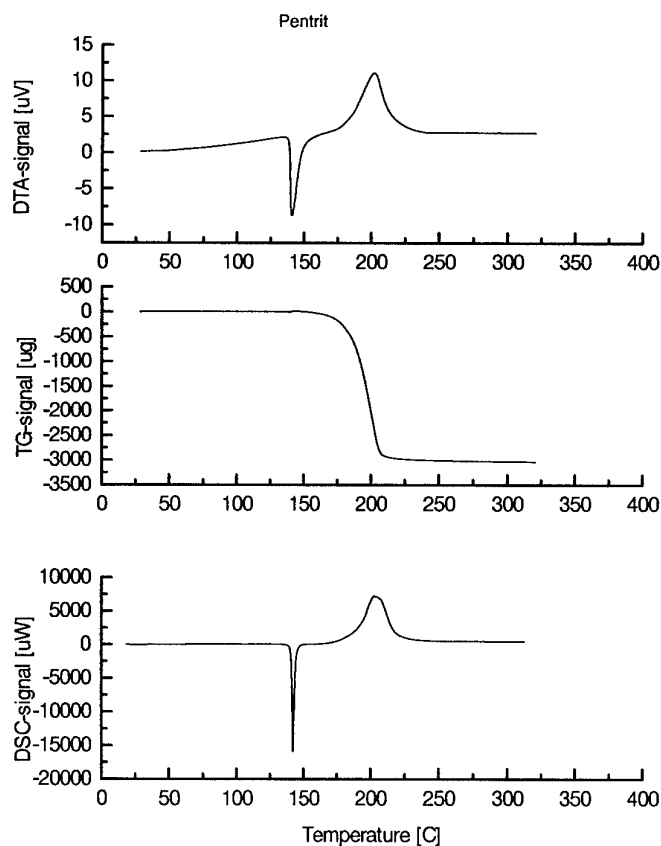
DSC		DTA/TG		Effect
Tmin	144/144	Tmin	144/145	Melting transition peak maximum
Tmax	202/205	Tmax	202/203	Decomposition exotherm peak max.
Tmelting 1	141/141	Tmelting	139/139	Onset of melting
Tonset	159/160	Tonset	165/164	Decomposition exotherm onset
Te onset	187/185	Te onset	179/181	Extrapolated onset
		Tmass loss 1	150/151	Start decomposition
		Mass loss 1	100/100	Mass loss
		Tmass loss 2	-/-	
		Mass loss 2	-/-	
Endoth. Enthalpy	154/153	Endoth. Enthalpy	132/127	Melting enthalpy
Exoth. Enthalpy	588/539	Exoth. Enthalpy	446/463	Sublimation enthalpy, by DTA

The decomposition of pentrit is observed as a one-step reaction.

A distinct endothermic melting peak occurs before the strong exothermic decomposition peak of PETN appears.

PETN starts to decompose at 151 °C (TG), its weight loss occurs relatively slowly.

The values for the melting points correspond with the literature values (Ref. 7, 9, 11).



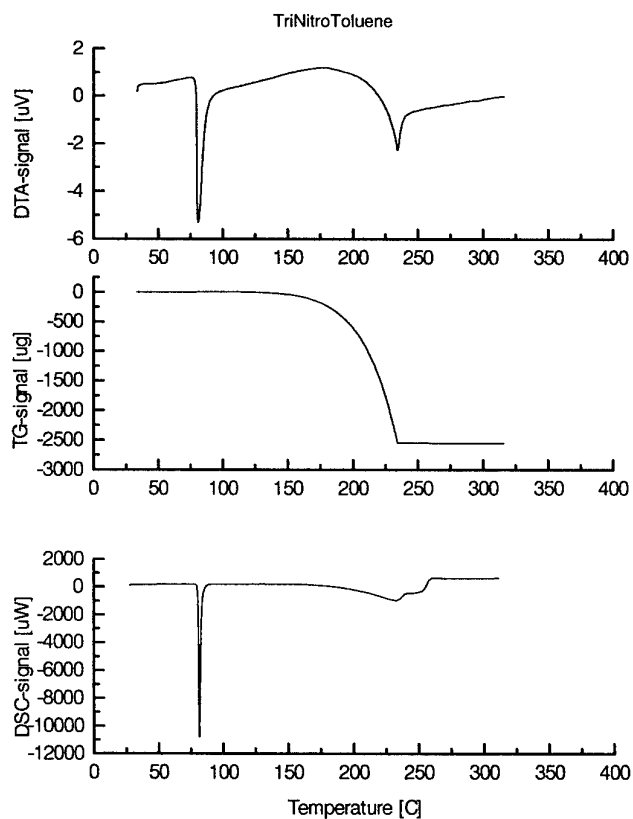
TNT - Trinitrotoluene - sample 94 EMK 021

DSC		DTA/TG		Effect
Tmin1	82/83	Tmin1	81/81	Melting transition peak maximum
Tmin2		Tmin2		Melting transition peak maximum
Tmax		Tmax		Decomposition exotherm peak max.
Tmelting 1	80/80	Tmelting 1	79/79	Onset of melting
Tmelting 2		Tmelting 2		Onset of melting
Tonset	233/245	Tonset	235/240	Decomposition exotherm onset
Te onset		Te onset		Extrapolated onset
		Tmass loss 1	125/126	Start decomposition
		Mass loss 1	100/100	Mass loss 1
		Tmass loss 2		Start decomposition
		Mass loss 2		Mass loss 2
		Total mass loss	100/100	
Endoth. Enthalpy 1	101/102	Endoth. Enthalpy 1	126/121	Melting enthalpy
Endoth. Enthalpy 2	238/180	Endoth. Enthalpy 2	94/106	Melting enthalpy
Exoth. Enthalpy		Exoth. Enthalpy		Sublimation enthalpy, by DTA

The decomposition of TNT is observed as a one-step reaction as measured by TG, with two endothermic effects in the DTA signal. One endotherm is due to the melting of TNT. The first endothermic enthalpy observed by DTA is difficult to determine.

The measured DTA values are in good agreement with the literature values (Ref. 7 and 9).

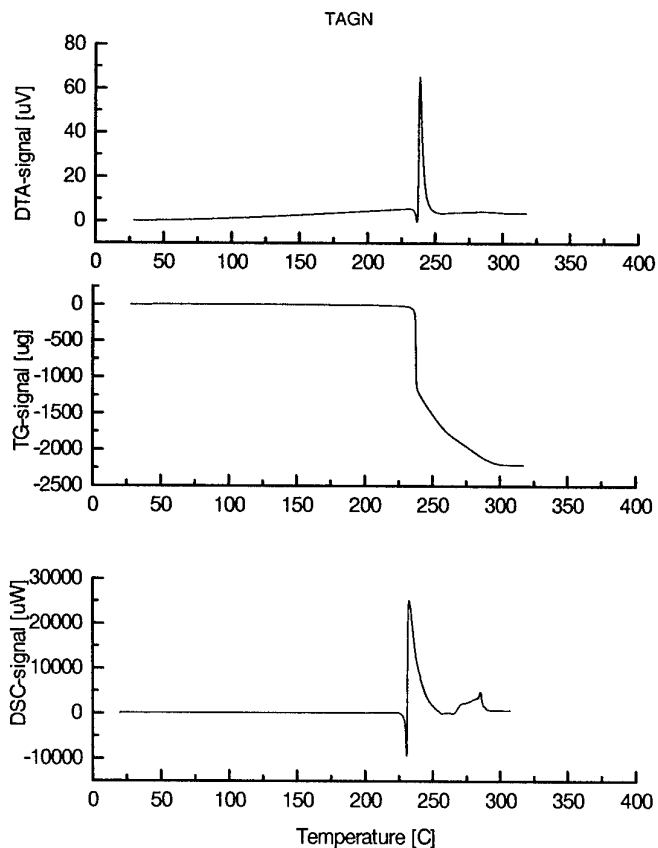
The duplicates of the second endothermic enthalpy differ, caused by the decomposition (evaporation) in the same temperature range.



TAGN - Triaminoguanidine Nitrate - sample 94 ACX 189

DSC		DTA/TG		Effect
Tmin	231/233	Tmin	236/236	Melting transition peak maximum
Tmax	233/234	Tmax	238/238	Decomposition exotherm peak max.
Tmelting 1	229/228	Tmelting	229/227	Onset of melting
Tonset	231/233	Tonset	232/232	Decomposition exotherm onset
Te onset	231/233	Te onset	-/-	Extrapolated onset
		Tmass loss 1	226/228	Start decomposition
		Mass loss 1	51/60	Mass loss
		Tmass loss 2	239/237	
		Mass loss 2	45/36	
		Total mass loss	96/96	
Endoth. Enthalpy	35/48	Endoth. Enthalpy	31/31	Melting enthalpy
Exoth. Enthalpy	760/775	Exoth. Enthalpy	600/523	Sublimation enthalpy, by DTA

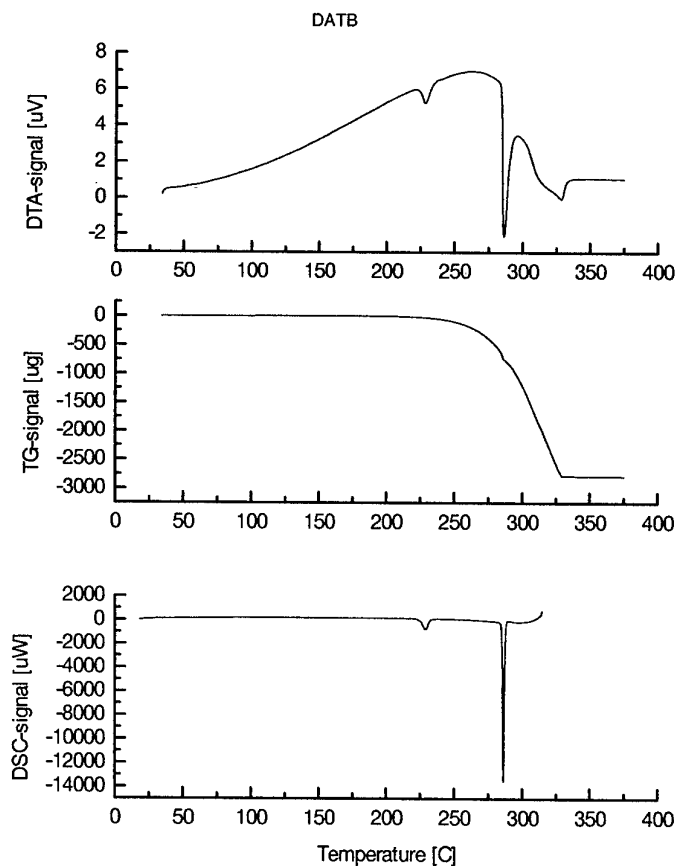
The exothermic reaction occurs immediately after the endothermic phase change from solid into liquid. As shown in the next figure, the TG curve consists of two steps. The first step corresponds to a rapid reaction also observed in the DTA curve between the weight loss of 0% (226 °C) and between ~ 55% (238 °C). The second stage is a slow weight loss from ~ 55% to 96% at 350 °C. The measured DTA values are in good agreement with the literature values (Ref. 7, 9 and 18).



DATB - Diaminotrinitro Benzene - sample 94 ACX 142

DSC		DTA/TG		Effect
Tmin1	229/228	Tmin1	226/225	Melting transition peak maximum
Tmin2	286/286	Tmin2	284/284	Melting transition peak maximum
Tmax	340/340	Tmax	-/-	Decomposition exotherm peak max.
Tmelting 1	225/225	Tmelting 1	222/223	Crystal transform.
Tmelting 2	285/285	Tmelting 2	283/283	Onset of melting
Tonset	302/306	Tonset	-/-	Decomposition exotherm onset
Te onset	-/-	Te onset	-/-	Extrapolated onset
		Tmass loss 1	213/212	Start decomposition
		Mass loss 1	100/100	Mass loss
		Tmass loss 2		
		Mass loss 2		
		Total mass loss	100/100	
Endoth. Enthalpy 1	17/18	Endoth. Enthalpy 1	12/13	Melting enthalpy
Endoth. Enthalpy 2	114/112	Endoth. Enthalpy 2	65/65	Melting enthalpy
Exoth. Enthalpy	-484	Exoth. Enthalpy	-/-	Sublimation enthalpy, by DTA

The decomposition of DATB is observed as a one-step reaction in the TG curve. The measured DTA values are in agreement with the literature values (Ref. 7), especially the temperature for the crystal transformation and the melting temperature. The values for the enthalpy (crystal transformation) are a little larger, but these differences are within the accuracy of the experiment. A solution for this is to measure a larger mass in a specified temperature range.



HEXAL - mixture of RDX and aluminium - sample 95 ACX 031

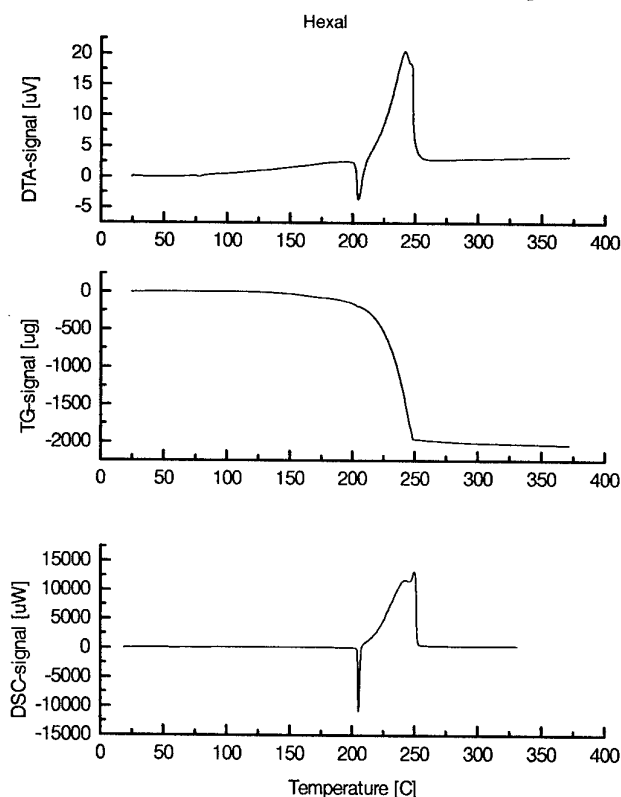
DSC		DTA/TG		Effect
Tmin1	205/205	Tmin1	204/205	Melting transition peak maximum
Tmin2		Tmin2		Melting transition peak maximum
Tmax	249/249	Tmax	239/242	Decomposition exotherm peak max.
Tmelting 1	204/204	Tmelting 1	203/204	Onset of melting
Tmelting 2		Tmelting 2		Onset of melting
Tonset	209/208	Tonset	212/212	Decomposition exotherm onset
Te onset	215/216	Te onset		Extrapolated onset
		Tmass loss 1	128/128	Start decomposition
		Mass loss 1	49/52	Mass loss 1
		Tmass loss 2		Start decomposition
		Mass loss 2		Mass loss 2
		Total mass loss	49/52	
Endoth. Enthalpy 1	68/51	Endoth. Enthalpy 1	38/40	Melting enthalpy
Endoth. Enthalpy 2		Endoth. Enthalpy 2		Melting enthalpy
Exoth. Enthalpy	961/667	Exoth. Enthalpy	554/349	Sublimation enthalpy, by DTA

The decomposition of HEXAL is observed as a one-step reaction, measured by TG.

Reference values are not available, only the value for the melting point of RDX could be used. Pure RDX has a melting point of 204 °C, so the value for Hexal is in agreement.

The remaining mass could be an indication of the percentage of aluminium, about 50%. A reference for this value is not available.

The exothermic enthalpies differ because of the inhomogeneity of the sample and the evaporation of RDX in the investigated temperature range.



NTO - 3-nitro-1,2,4-triazol-5-one - sample 95 ACX 176

DSC		DTA/TG		Effect
Tmin1		Tmin1		Melting transition peak maximum
Tmin2		Tmin2		Melting transition peak maximum
Tmax	272/273	Tmax	274/272	Decomposition exotherm peak max.
Tmelting 1		Tmelting 1		Onset of melting
Tmelting 2		Tmelting 2		Onset of melting
Tonset	252/254	Tonset	255/258	Decomposition exotherm onset
Te onset	268/269	Te onset	264/267	Extrapolated onset
		Tmass loss 1	216/216	Start decomposition
		Mass loss 1	40/18	Mass loss 1
		Mass loss 2	228/228	Start decomposition
		Mass loss 2	51/71	Mass loss 2
		Total mass loss	91/89	
Endoth. Enthalpy 1		Endoth. Enthalpy 1		Melting enthalpy
Endoth. Enthalpy 2		Endoth. Enthalpy 2		Melting enthalpy
Exoth. Enthalpy	635/716	Exoth. Enthalpy	328/345	Sublimation enthalpy, by DTA

The decomposition of NTO is observed as a two-step reaction by TG.

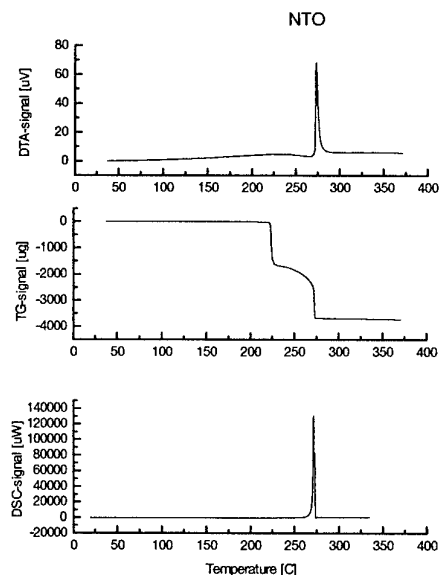
The DSC spectrum of NTO showed only one peak; a very strong exothermic peak at 235 °C. An extrapolated onset temperature of 270 °C has also been reported in Ref. 12.

By DSC, no endothermic transitions were observed on heating NTO. An exothermic extrapolating temperature of 268 °C and a broad shoulder beginning at 232 °C was observed.

The residual solid (at 350 °C) contains roughly 10% of the original NTO mass, this is in good agreement with Ref. 12.

The two stages in the TG curve are caused by an impurity in the investigated NTO-sample. Other analysis has shown that the used sample was polluted with nitric acid and resulted in a low pH-value. Impurities can result in a different thermogram than would be obtained with a pure chemical substance.

The resulting values of the acidity analysis and the thermogram are not in agreement with the specifications in the STANAG (Ref. 3).



AP - Ammonium perchlorate - sample 95 EMK 023

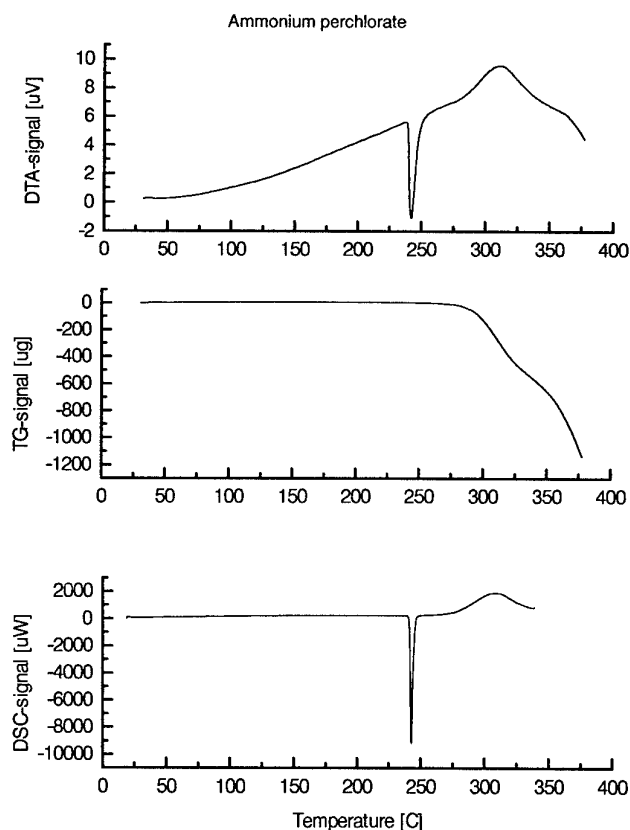
DSC		DTA/TG		Effect
Tmin1	243/243	Tmin1	243/242	Melting transition peak maximum
Tmin2		Tmin2		Melting transition peak maximum
Tmax	309/313	Tmax	312/313	Decomposition exotherm peak max.
Tmelting 1	242/241	Tmelting 1	238/238	Phase transition
Tmelting 2		Tmelting 2		Onset of melting
Tonset	256/257	Tonset	261/262	Decomposition exotherm onset
Te onset	275/278	Te onset		Extrapolated onset
		Tmass loss 1	281/280	Start decomposition
		Mass loss 1	21/21	Mass loss 1
		Tmass loss 2	354/354	Start decomposition
		Mass loss 2	27/28	Mass loss 2
		Total mass loss	48/49	
Endoth. Enthalpy 1	79/82	Endoth. Enthalpy 1	66/68	Melting enthalpy
Endoth. Enthalpy 2		Endoth. Enthalpy 2		Melting enthalpy
Exoth. Enthalpy	130/159	Exoth. Enthalpy	206/226	Sublimation enthalpy, by DTA

The decomposition of ammonium perchlorate is observed as a two-step reaction by TG.

Decomposition of AP depends strongly on the added catalyst (Ref. 13) and the particle size.

The endothermic peak can be attributed to the phase transition of AP from the orthorhombic to the cubic form.

The measured DTA values are in good agreement with the literature values (Ref. 8).

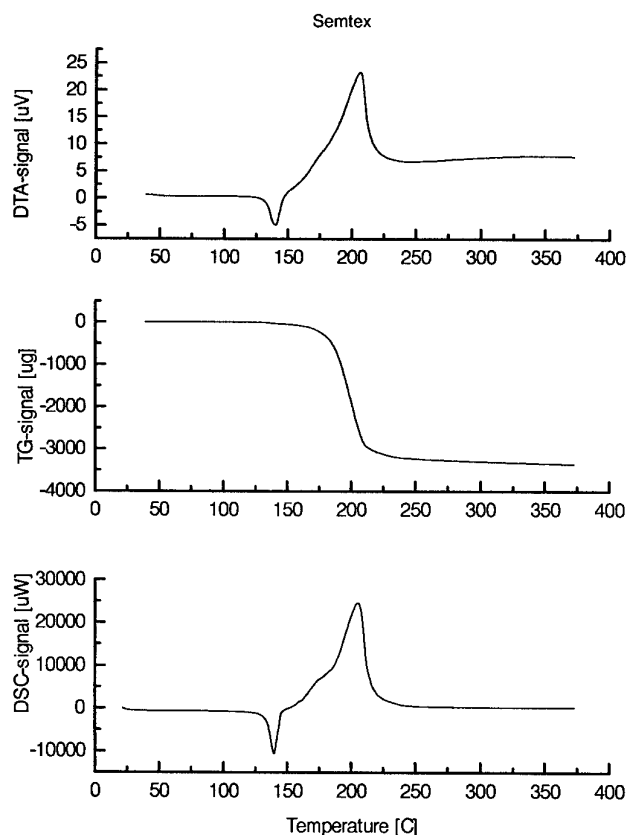


Semtex - sample 94 ACX 170

DSC		DTA/TG		Effect
Tmin1	141/139	Tmin1	141/144	Melting transition peak maximum
Tmin2		Tmin2		Melting transition peak maximum
Tmax	202/205	Tmax	202/202	Decomposition exotherm peak max.
Tmelting 1	134/134	Tmelting 1	132/135	Onset of melting
Tmelting 2		Tmelting 2		Onset of melting
Tonset	154/154	Tonset	147/152	Decomposition exotherm onset
Te onset	177/173	Te onset	167/171	Extrapolated onset
		Tmass loss 1	131/131	Start decomposition
		Mass loss 1	95/95	Mass loss 1
		Tmass loss 2		Start decomposition
		Mass loss 2		Mass loss 2
		Total mass loss		
Endoth. Enthalpy 1	117/117	Endoth. Enthalpy 1	76/78	Melting enthalpy
Endoth. Enthalpy 2		Endoth. Enthalpy 2		Melting enthalpy
Exoth. Enthalpy	1053/1259	Exoth. Enthalpy	1001/1130	Sublimation enthalpy, by DTA

The decomposition of Semtex is observed as a one-step reaction, measured by TG. Semtex is a plastic explosive formulated on the basis of PETN (Ref. 14). According to this information, the start of the weight loss is at 131 °C. The resulting melting point in the curves indicates that the endothermic effect belongs to the melting of PETN (see results of pure PETN).

Based on the endothermic enthalpy from the DSC measurements it is possible to calculate the percentage of PETN. In the investigated sample, the percentage of PETN is about 73%. A reference for this value is not available.



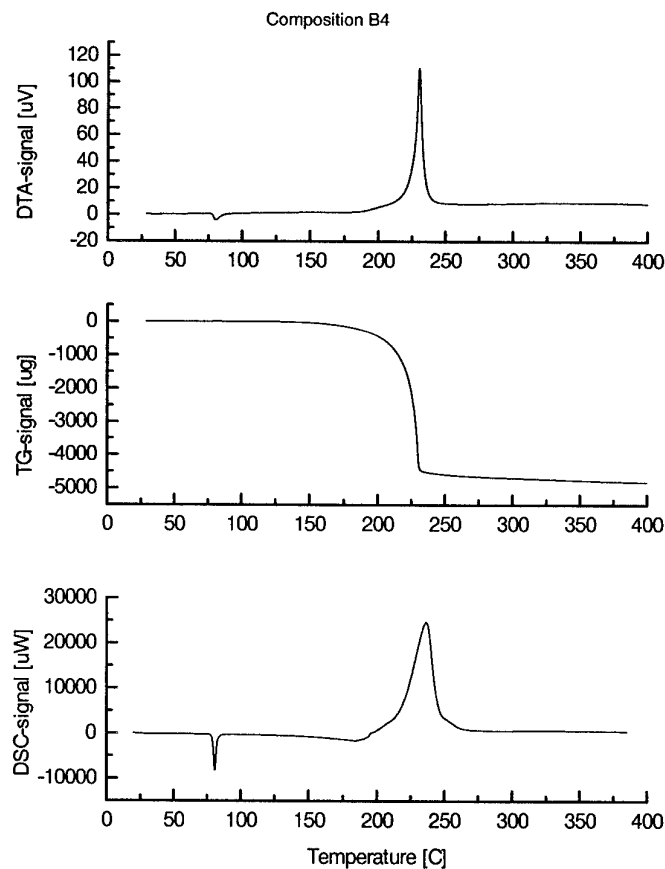
Composition B4 - sample 95 EMK 019

DSC		DTA/TG		Effect
Tmin1	81/81	Tmin1	80/81	Melting transition peak maximum
Tmin2		Tmin2		Melting transition peak maximum
Tmax	237/236	Tmax	229/231	Decomposition exotherm peak max.
Tmelting 1	79/79	Tmelting 1	78/78	Onset of melting
Tmelting 2		Tmelting 2		Onset of melting
Tonset	186/183	Tonset	184/185	Decomposition exotherm onset
Te onset	216/216	Te onset	222/223	Extrapolated onset
		Tmass loss 1	132/133	Start decomposition
		Mass loss 1	93/95	Mass loss 1
		Tmass loss 2		Start decomposition
		Mass loss 2		Mass loss 2
		Total mass loss		
Endoth. Enthalpy 1	40/41	Endoth. Enthalpy 1	29/32	Melting enthalpy
Endoth. Enthalpy 2		Endoth. Enthalpy 2		Melting enthalpy
Exoth. Enthalpy	1056/1020	Exoth. Enthalpy	1013/980	Sublimation enthalpy, by DTA

The decomposition of composition B4 is observed as a one-step reaction.

Calculation of the percentage of TNT is possible on the basis of the endothermic enthalpy (melting) of TNT, namely 99 kJ/kg, results in 40% TNT. The resulting mass of about 5% after the TG experiment is probably from the additives in Composition B4.

The measured values are in good agreement with the literature values (Ref. 7 and 9) for the mixture of RDX and TNT.

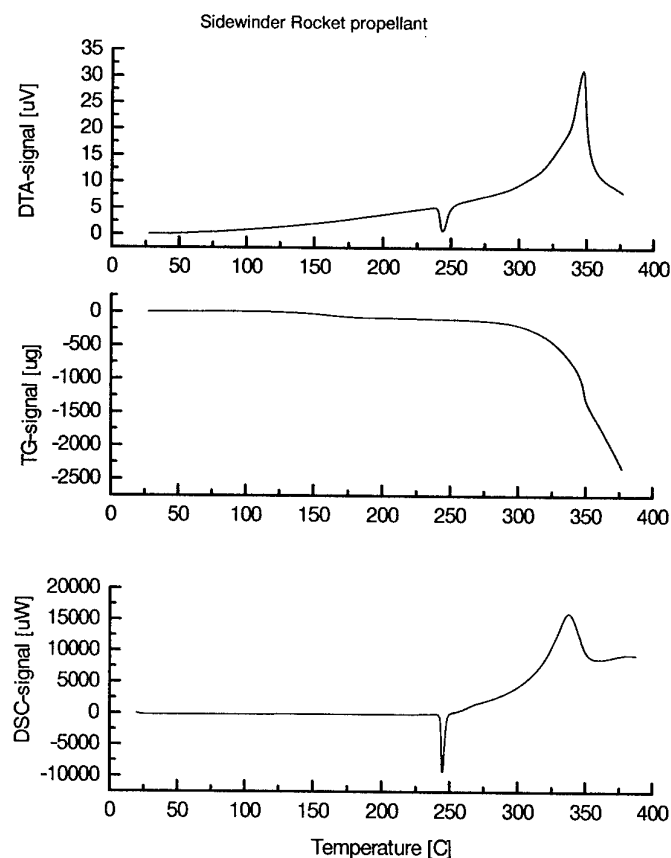


Sidewinder (AP - HTPB) - sample 95 ACX 033-2

DSC		DTA/TG		Effect
Tmin1	245/245	Tmin1	244/244	Melting transition peak maximum
Tmin2		Tmin2		Melting transition peak maximum
Tmax	338/349	Tmax	347/348	Decomposition exotherm peak max.
Tmelting 1	243/243	Tmelting 1	239/239	Onset of melting
Tmelting 2		Tmelting 2		Onset of melting
Tonset	280/286	Tonset	266/267	Decomposition exotherm onset
Te onset	313/319	Te onset		Extrapolated onset
		Tmass loss 1	130/128	Start decomposition
		Mass loss 1	2.9/3.0	Mass loss 1
		Tmass loss 2	220/220	Start decomposition
		Mass loss 2	88/94	Mass loss 2
		Total mass loss	91/97	
Endoth. Enthalpy 1	67/68	Endoth. Enthalpy 1	60/56	Melting enthalpy
Endoth. Enthalpy 2		Endoth. Enthalpy 2		Melting enthalpy
Exoth. Enthalpy	535/845	Exoth. Enthalpy	958/1091	Sublimation enthalpy, by DTA

The decomposition of a rocket propellant sample of the Sidewinder is observed as a one-step reaction by TG.

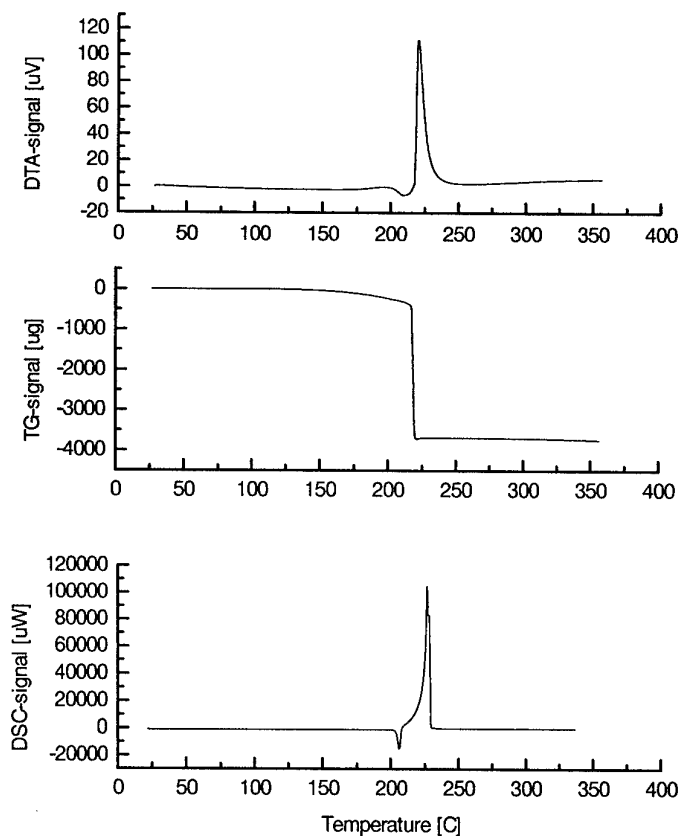
The initial 3% mass loss can be explained by the evaporation of IDPI, which is present for 3% the original sample. The start of decomposition of HTPB by TG starts above a temperature of 320 °C, Ref. 15.



PBX - RU - 113

DSC		DTA/TG		Effect
Tmin1	206/206	Tmin1	210/210	Melting transition peak maximum
Tmin2		Tmin2		Melting transition peak maximum
Tmax	227/227	Tmax	221/221	Decomposition exotherm peak max.
Tmelting 1	204/204	Tmelting 1	200/202	Onset of melting
Tmelting 2		Tmelting 2		Onset of melting
Tonset	209/209	Tonset	217/217	Decomposition exotherm onset
Te onset	210/210	Te onset		Extrapolated onset
		Tmass loss 1	117/119	Start decomposition
		Mass loss 1	8/8	Mass loss 1
		Tmass loss 2	212/212	Start decomposition
		Mass loss 2	77/78	Mass loss 2
		Total mass loss	85/86	
Endoth. Enthalpy 1	97/99	Endoth. Enthalpy 1		Melting enthalpy
Endoth. Enthalpy 2		Endoth. Enthalpy 2		Melting enthalpy
Exoth. Enthalpy	1026/996	Exoth. Enthalpy	501/509	Sublimation enthalpy, by DTA

The decomposition of the PBX RU-113 is observed as a two-step reaction. The first stage is the evaporation of IDPI (~8%), followed by the decomposition of the RDX. The remaining mass of 15% is HTPB. Pure HTPB decomposes from a temperature of 300 °C. The mass loss up to 350 °C is only about 4% (Ref. 15). So it indicates that after a strong decrease in weight, the remaining mass belongs to HTPB.

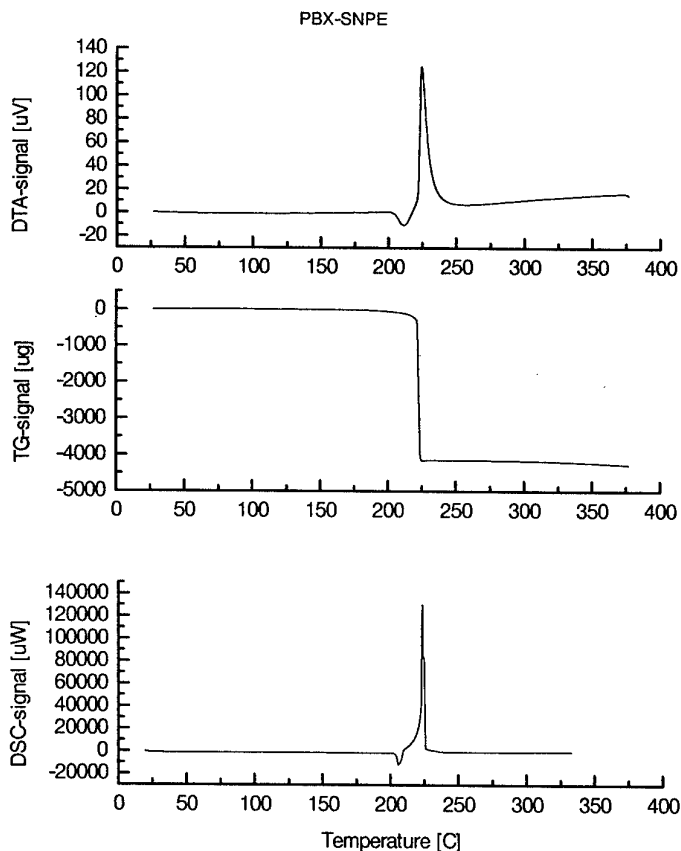


PBX - SNPE

<i>DSC</i>		<i>DTA/TG</i>		<i>Effect</i>
Tmin1	206/206	Tmin1	212/210	Melting transition peak maximum
Tmin2		Tmin2		Melting transition peak maximum
Tmax	224/226	Tmax	225/224	Decomposition exotherm peak max.
Tmelting 1	204/204	Tmelting 1	204/204	Onset of melting
Tmelting 2		Tmelting 2		Onset of melting
Tonset	210/209	Tonset	218/217	Decomposition exotherm onset
Te onset	211/211	Te onset		Extrapolated onset
		Tmass loss 1	193/192	Start decomposition
		Mass loss 1	85/85	Mass loss 1
		Tmass loss 2		Start decomposition
		Mass loss 2		Mass loss 2
		Total mass loss		
Endoth. Enthalpy 1	83/89	Endoth. Enthalpy 1		Melting enthalpy
Endoth. Enthalpy 2		Endoth. Enthalpy 2		Melting enthalpy
Exoth. Enthalpy	853/1033	Exoth. Enthalpy	495/422	Sublimation enthalpy, by DTA

The decomposition of the PBX SNPE is observed as a one-step reaction. The mass decrease is the decomposition of the RDX. The remaining mass of 15% is HTPB. Pure HTPB decomposes from a temperature of 300 °C. The mass loss up to 350 °C is only about 4%. So it indicates that after a strong decrease in weight, the remaining mass belongs to HTPB.

Here no effect of the initial IDPI is observed, probably another compound has been used.



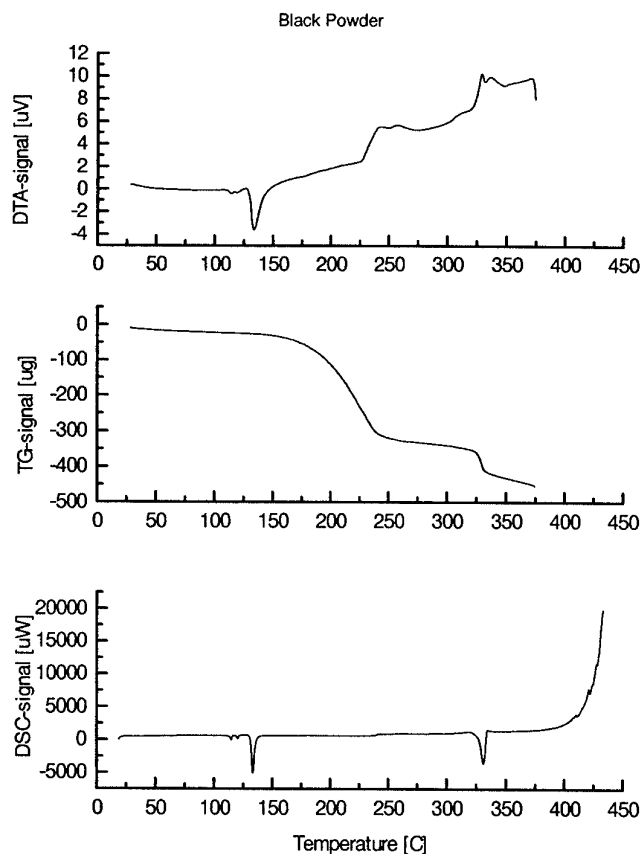
Black powder

DSC		DTA/TG		Effect
Tmin1	115/115	Tmin1		Melting transition peak maximum
Tmin2	120/120	Tmin2		Melting transition peak maximum
Tmin3	133/133	Tmin3	134/134	Melting transition peak maximum
Tmelting 1		Tmelting 1		Onset of melting
Tmelting 2		Tmelting 2		Onset of melting
Tonset		Tonset		Decomposition exotherm onset
Te onset	-/381	Te onset		Extrapolated onset
		Tmass loss 1	150/150	Start decomposition
		Mass loss 1	10/9	Mass loss 1
		Tmass loss 2	300/300	Start decomposition
		Mass loss 2	4/4	Mass loss 2
		Total mass loss	13/13	
Endoth. Enthalpy 1		Endoth. Enthalpy 1		Melting enthalpy
Endoth. Enthalpy 3	36/38	Endoth. Enthalpy 3	26/26	Melting enthalpy
Exoth. Enthalpy		Exoth. Enthalpy		Sublimation enthalpy, by DTA

The decomposition of black powder is observed as a two-step reaction.

The measured values are in good agreement with the literature values (Ref. 7 and 16).

In the region 110 -140 °C, the individual peaks are due to KNO₃ and S. The first two endotherms belong to sulphur and the third peak to KNO₃. The onset temperature belongs to the decomposition of the charcoal. The mass loss in the TG curve is caused by the decomposition of the complete mixture.



6 Thermal Analysis studies on propellants

6.1 Sample mass of investigated propellants

According to STANAG 4515 (Ref. 5), it is recommended to weigh the sample to an accuracy of 0.1 mg. The mass of the sample weights are given in Table 8. The weight of the sample cup including the sample is measured before and after the experiment, to calculate the loss of weight, only for the DSC measurements.

Table 8: *Sample mass of the propellants.*

Sample number	Number of propellant	Mass of sample DTA/TG [milligram]	Mass of sample DSC [milligram]
P - 1	KB 5494	3.395/5.109	1.922/1.738
P - 2	KB 6856	3.438/3.791	2.213/2.235
P - 3	KB 6971	4.909/4.361	2.065/2.178
P - 4	KB 6981	6.023/5.765	3.918/3.489
P - 5	KB 7057	5.598/5.804	2.313/3.695

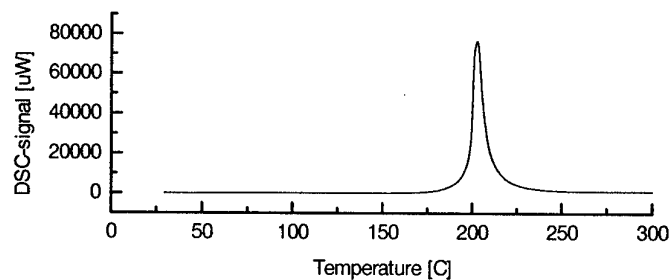
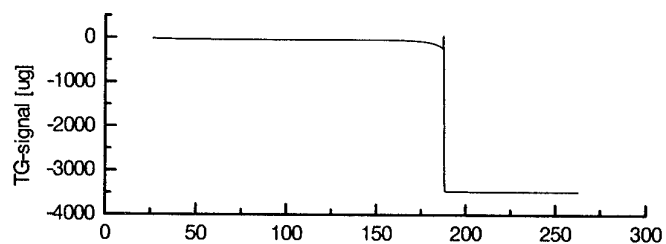
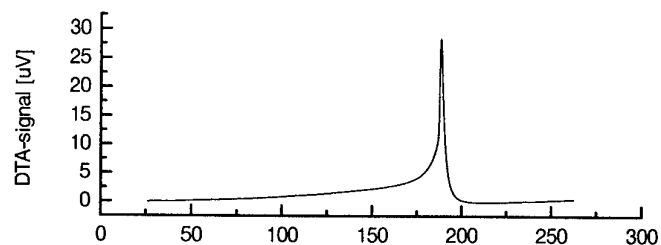
6.2 Thermograms of the investigated propellants

On the following pages, the results of the different propellants are presented in tabular form, followed by figures of the DSC and TG/DTA measurements. See page 4 for an explanation of the abbreviations used.

KB 6856 - Single-base propellant

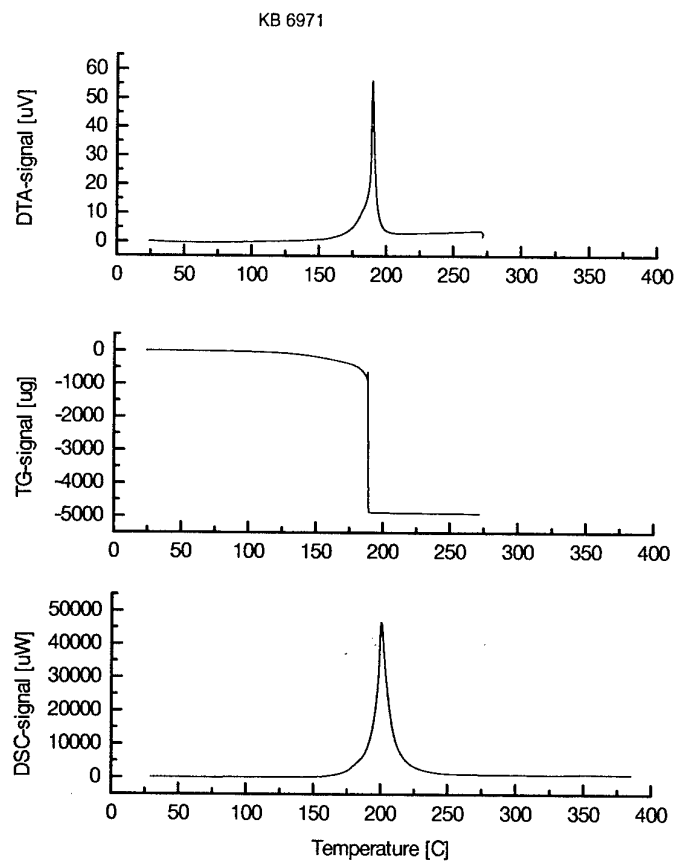
DSC		DTA/TG		Effect
Tmax1	203/203	Tmax1	188/190	Decomposition exotherm peak max.
Tonset	170/171	Tonset	162/162	Decomposition exotherm onset
Te onset	198/198	Te onset	185/187	Extrapolated onset
		Tmass loss 1	168/170	Start decomposition
		Mass loss 1	100/100	Mass loss 1
Exoth. Enthalpy	2231/2173	Exoth. Enthalpy.	275/275	Sublimation enthalpy, by DTA

KB 6856



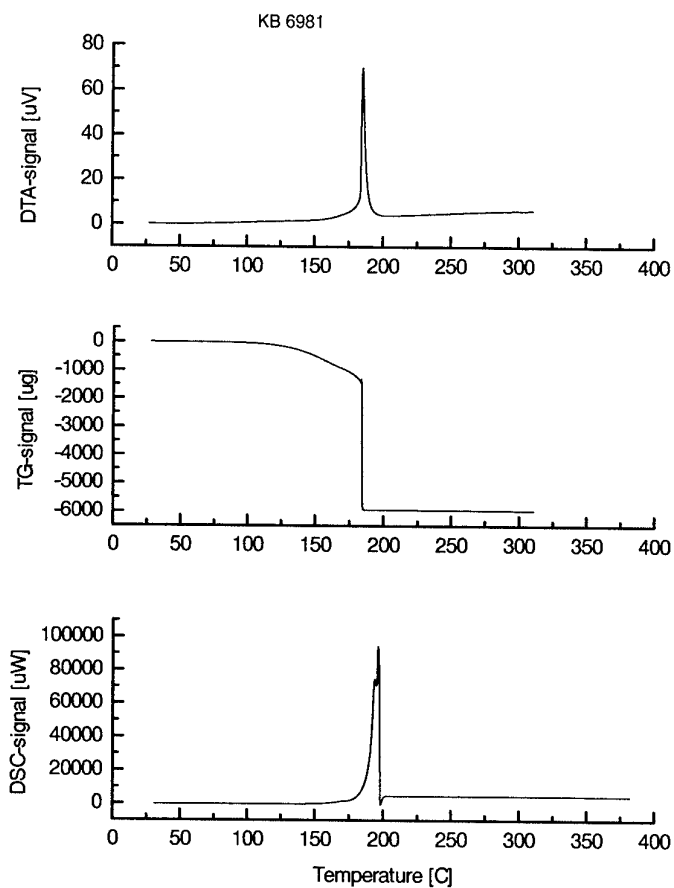
KB 6971 - Double-base propellant

DSC		DTA/TG		Effect
Tmax1	200/200	Tmax1	190/189	Decomposition exotherm peak max.
Tmax2		Tmax2		Decomposition exotherm peak max.
Tonset	155/154	Tonset	126/125	decomposition exotherm onset
Te onset	195/195	Te onset	184/183	Extrapolated onset
		Tmass loss 1	101/98	Start decomposition
		Mass loss 1	100/100	Mass loss 1
Exoth. Enthalpy	1951/1936	Exoth. Enthalpy	377/438	Sublimation enthalpy, by DTA



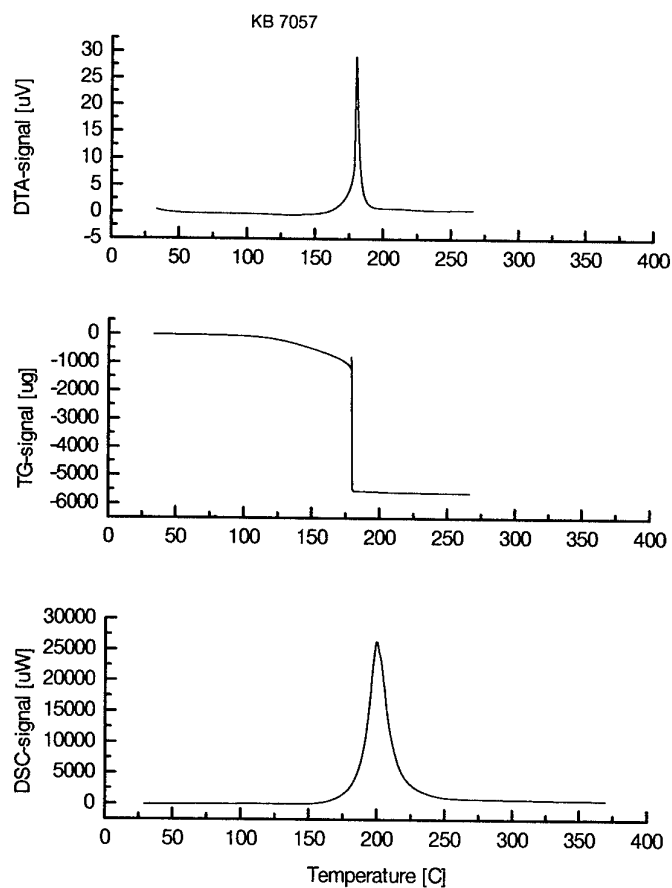
KB 6981 - Double-base propellant

DSC		DTA/TG		Effect
Tmax1	196/195	Tmax1	184/182	Decomposition exotherm peak max.
Tmax2		Tmax2		Decomposition exotherm peak max.
Tonset	154/155	Tonset	145/148	Decomposition exotherm onset
Te onset	189/190	Te onset	182/181	Extrapolated onset
		Tmass loss 1	95/95	Start decomposition
		Mass loss 1	99/99	Mass loss 1
Exoth. Enthalpy	825/1493	Exoth. Enthalpy	281/285	Sublimation enthalpy, by DTA



KB 7057 - DEGN propellant

DSC		DTA/TG		Effect
Tmax1	200/200	Tmax1	180/179	Decomposition exotherm peak max.
Tmax2		Tmax2		Decomposition exotherm peak max.
Tonset	153/153	Tonset	150/153	Decomposition exotherm onset
Te onset		Te onset	178/177	Extrapolated onset
		Tmass loss 1	94/95	Start decomposition
		Mass loss 1	100/100	Mass loss 1
Exoth. Enthalpy	1456/1444	Exoth. Enthalpy	154/228	Sublimation enthalpy, by DTA



KB 5494 - Triple-base propellant

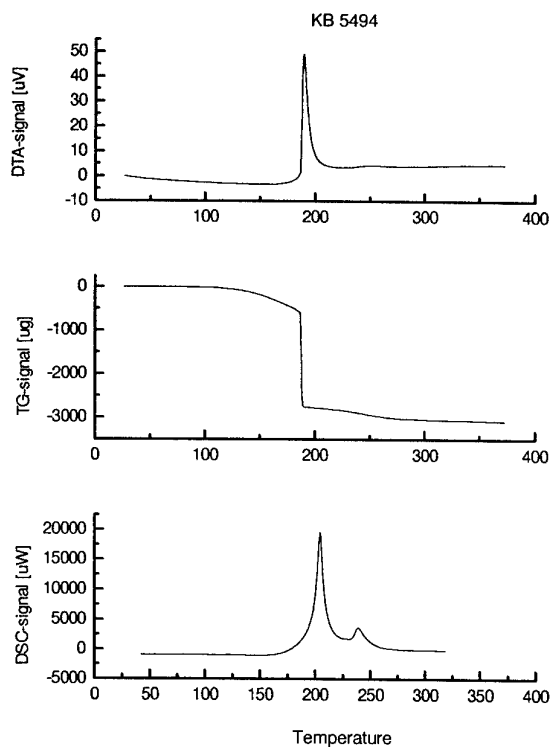
DSC		DTA/TG		Effect
Tmax1	207/206	Tmax1	187/185	Decomposition exotherm peak max.
Tmax2	239/238	Tmax2		Decomposition exotherm peak max.
Tonset	162/160	Tonset	164/162	Decomposition exotherm onset
Tonset2	232/231	Tonset2		Decomposition exotherm onset
Te onset		Te onset		Extrapolated onset
Te onset2		Te onset2		Extrapolated onset
		Tmass loss 1	96/93	Start decomposition
Exoth. Enthalpy	620/612	Mass loss 1	90/92	Mass loss 1
Exoth. Enthalpy 2	44/44	Exoth. Enthalpy	330/286	
Exoth. Enthalpy total	1165/1170	Exoth. Enthalpy 2		

All the DSC curves show one sharp exothermic peak, except for the curve of KB 5494, the triple-base propellant, which shows two peaks.

An explanation for this effect may be found in the composition of the propellant (e.g. the content of nitroglycerine).

Looking at the maximum peak temperatures, it can be concluded that the variation is very small. The propellant with the highest nitroglycerine content has the lowest value. Nitroglycerine decreases the maximum peak temperature or it decreases the decomposition rate.

The determination of the exothermic enthalpy by DTA is only useful as an indication. An exact determination of the enthalpy is only possible by a quantitative technique, namely DSC, especially with closed stainless steel cups.



The TG curves could be divided into an initial step, up to ~ 185 °C, and a fast second step, to a mass loss maximum at ~ 195 °C, except for KB 7057. A difference in starting temperature is measured, caused by the composition of the propellant.

The mass loss is presented in Table 9.

Table 9: *Mass loss of the propellants.*

KB 5494	88 - 0%	185 - 16.4%	195 - 82%	287 - 90%
KB 6856	152 - 0%	186 - 6%	195 - 100%	
KB 6971	118 - 0%	187 - 15%	194 - 100%	
KB 6981	86 - 0%	181 - 21%	191 - 99%	
KB 7057	96 - 0%	177 - 19%	184 - 99%	

After the TG measurements, the residual weight is mostly nearly zero, except the triple-base propellant, so the decomposition is about 100%. The DSC measurements give another result, namely;

KB 6856 $\Rightarrow \sim 15\%$

KB 6971 $\Rightarrow \sim 14\%$

KB 6981 $\Rightarrow \sim 15\%$

KB 5494 $\Rightarrow \sim 25\%$

KB 7057 $\Rightarrow \sim 9\%$

The values of mass loss for the SB and DB propellants are in the same range as the investigated propellants presented in Ref. 17. The differences between the propellants depend on the composition and the decomposition mode.

7 Thermal Analysis studies on polymers

7.1 Sample mass of investigated polymers

Table 10 lists the polymers used in the experiments for the compatibility testing of propellants with DSC and TG/DTA. The polymers did not have a known relation with practical situations in ammunition. In an earlier programme, Ref. 6, the same propellants were investigated by the Isothermal Storage Test and the Vacuum Stability Test.

Table 10: *Sample mass of the polymers.*

Sample number	Name of the polymer	Mass of sample DTA/TG [milligram]	Mass of sample DSC [milligram]
PL - 1	Nylon 6/6	5.825/4.433	5.427/4.272
PL - 2	Cellulose Acetate	4.492/4.193	3.582/3.079
PL - 3	Polyvinylchloride	3.858/4.546	3.852/3.160
PL - 4	PMMA	4.195/4.569	4.499/4.826

7.2 Thermograms of the investigated polymers

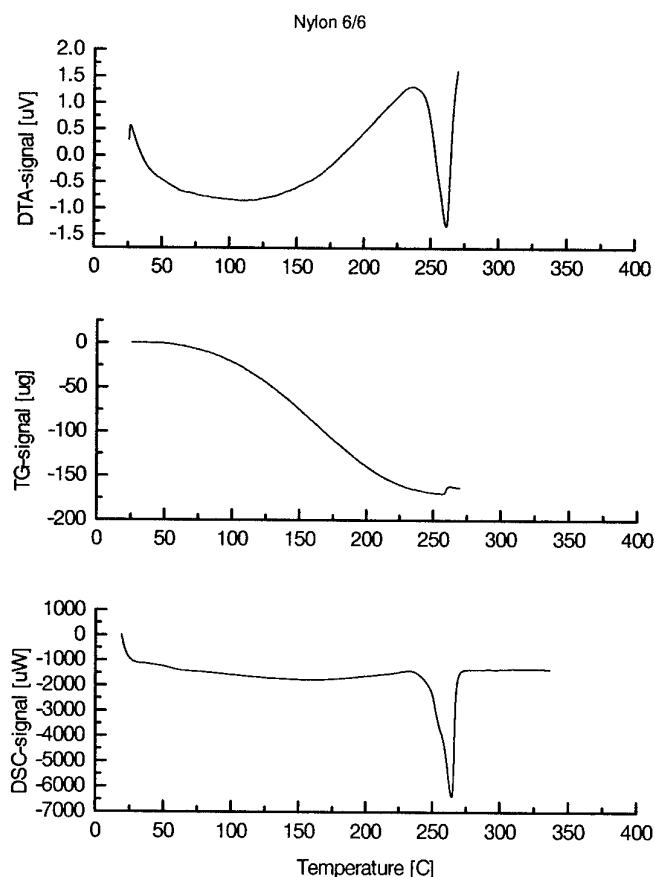
On the following pages, the results of the different polymers are presented in tabular form, followed by figures of the DSC and TG/DTA measurements. See page 4 for an explanation of the abbreviations used.

Nylon 6/6

DSC		DTA/TG		Effect
Tmax		Tmax		Decomposition exotherm peak max.
Tmin	265/265	Tmin	262/261	Melting transition peak maximum.
Tmelting	247/248	Tmelting	246/247	Onset of melting
Tonset		Tonset		Decomposition exotherm onset
Te onset		Te onset		Extrapolated onset
		Tmass loss 1	80/80	Start decomposition
		Mass loss 1	2.9/2.8	Mass loss 1
Endoth. Enthalpy	66/64	Endoth. Enthalpy	36/36	Melting enthalpy
Exoth. Enthalpy		Exoth. Enthalpy 2		

The curves of Nylon 6/6 show only a melting peak at a temperature of 247 °C. The TG signal results in a small mass decrease of 2.9% over the investigated temperature range.

The endothermic enthalpy could only be determined by DSC. The weight loss in the TG experiment disturbs the enthalpy determination.



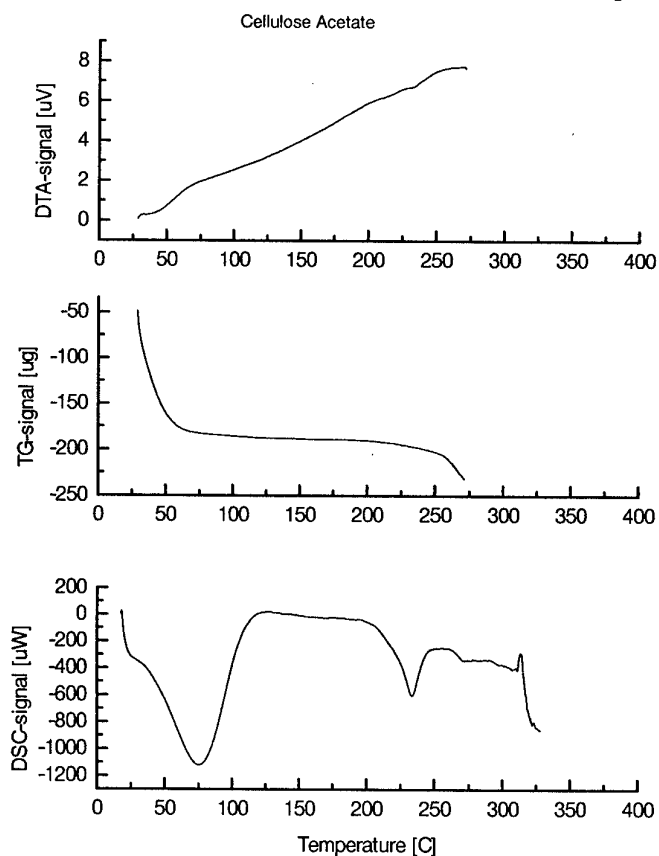
Cellulose Acetate

DSC		DTA/TG		Effect
Tmax		Tmax		Decomposition exotherm peak max.
Tmin	234/233	Tmin		Melting transition peak maximum.
Tmelting	216/216	Tmelting		Onset of melting
Tonset		Tonset		Decomposition exotherm onset
Te onset		Te onset		Extrapolated onset
		Tmass loss	30/30	Start decomposition
		Mass loss	4/4	Mass loss 1
Endoth. Enthalpy	12/10	Endoth. Enthalpy		Melting enthalpy
Exoth. Enthalpy		Exoth. Enthalpy		

In the DTA curve, no heat effects from cellulose acetate are detectable. The endotherm observed with DSC can therefore be interpreted as a melting peak of cellulose acetate.

The endothermic enthalpy due to the melting of cellulose acetate is very small.

Cellulose acetate is a stable polymer, up to 350 °C, except for melting.



PVC - Polyvinylchloride

DSC		DTA/TG		Effect
Tmax	267/267	Tmax		Decomposition exotherm peak max.
Tmin	92/91	Tmin		Melting transition peak maximum.
Tmelting	88/87	Tmelting		Onset of melting
Tonset	258/258	Tonset	150/150	Decomposition exotherm onset
Te onset	261/261	Te onset		Extrapolated onset
		Tmass loss	200/200	Start decomposition
Endoth. Enthalpy	2/2	Mass loss	16/16	Mass loss 1
Exoth. Enthalpy	995/1000	Endoth. Enthalpy		Melting enthalpy
		Exoth. Enthalpy		

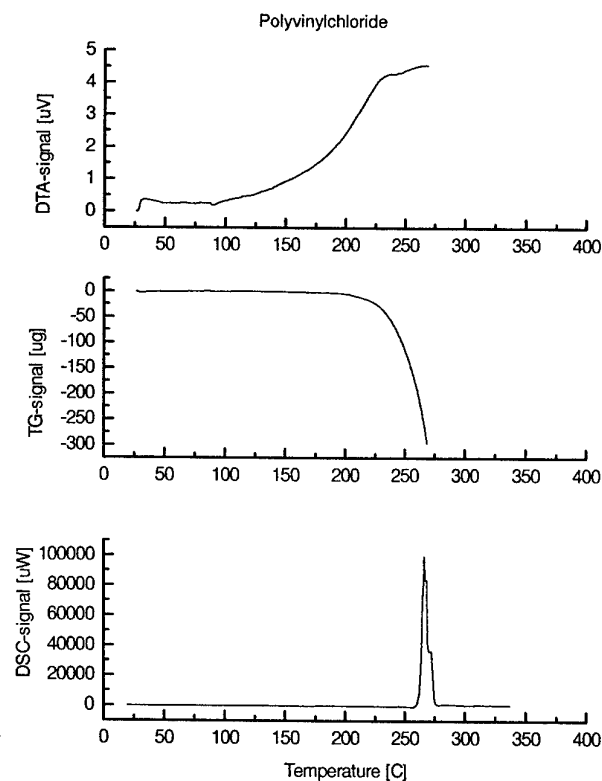
The melting enthalpy of PVC is very small, but when an exothermal reaction occurs, it is a fast reaction.

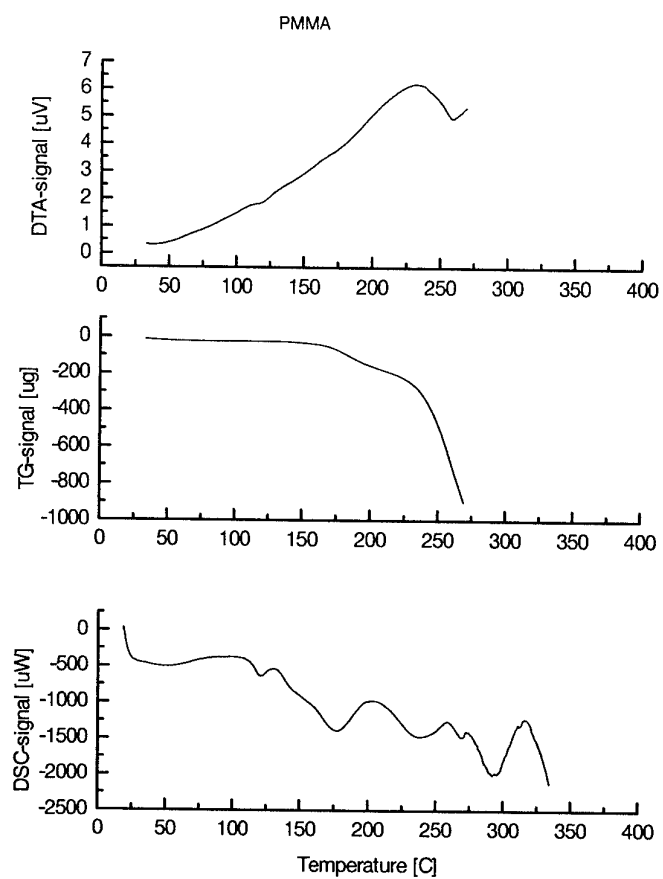
PMMA

PMMA shows no large heat effects; in the DSC equipment and the DTA/TG equipment only some small endothermal effects are measured by DSC.

The DTA signal shows a little exothermic reaction, with a small enthalpy, due to a reaction causing mass loss (see TG).

DSC		DTA/TG		Effect
		Tmass loss	115/125	start decomposition
		Mass loss	29/29	mass loss 1





8 Compatibility by DSC and DTA/TG

8.1 Theory according to STANAG 4147

The principle of STANAG 4147 (Ref. 4), is to provide evidence that a material may be used in an item of ammunition without detriment to the safety or reliability of an explosive with which it is in contact.

The STANAG describes a heating rate of 2 °C/min; in this test programme a heating rate of 10 °C has been used.

The criterium for compatibility by DSC is, the shift of the maximum peak temperature of no more than 4 °C.

For TG measurement, the weight loss of the mixture is not allowed to be more than 4% difference from the neat components. The calculation is to be performed at a fixed temperature.

8.2 Sample mass/ratio of the investigated mixtures

In the view of this project, only one propellant has been investigated, namely KB 6981, which is used in an earlier programme (Ref. 6). A comparison will be made between the obtained results.

In Table 11, the sample mass are presented for the measurements in DSC and in Table 12 for the measurements with DTA/TG.

Table 11: Sample mass for compatibility testing in DSC with KB 6981.

Sample number	Name of the polymer	Mass of sample propellant [milligram]	Mass of sample polymer [milligram]
PPL - 1	Nylon 6/6	3.259/3.506	1.810/3.655
PPL - 2	Cellulose Acetate	3.237/3.919	1.616/3.719
PPL - 3	Polyvinylchloride	3.981/3.671	1.759/3.201
PPL - 4	PMMA	3.508/3.393	3.333/1.590

Table 12: Sample mass for compatibility testing in DTA/TG with KB 6981.

Sample number	Name of the polymer	Mass of sample propellant [milligram]	Mass of sample polymer [milligram]
PPL - 1	Nylon 6/6	4.421/4.708	2.082/4.823
PPL - 2	Cellulose Acetate	4.014/3.985	2.263/3.763
PPL - 3	Polyvinylchloride	4.156/3.971	2.066/3.959
PPL - 4	PMMA	4.841/4.989	2.941/4.820

The measurements for the mixtures were carried out under the same conditions as the separate components.

8.3 Thermograms of the investigated mixtures

On the following pages, the results of the different combinations of KB6981 with polymers are presented in tabular form, followed by a figure of the DSC and TG/DTA measurement.

See page 4 an the explanation of the abbreviations used.

Table 13: Results of compatibility research by DSC.

Composition	Tmax	T extrapolated onset
Pure KB 6981	195 °C	189 °C
With nylon 6/6	193 °C	190 °C
With cell. acetate	194 °C	190 °C
With PVC	194 °C	190 °C
With PMMA	194 °C	190 °C

Based on the results of Table 13, it may be concluded that all the investigated combinations are compatible. The influence of the amount of polymer is negligible, because the main reaction is caused by the decomposition of the propellant.

Table 14: Results of compatibility research by TG at a temperature of 182 °C.

Composition	Mass decrease of TG test-pure	Mass decrease of TG test mixture	Calculated weight loss
Pure KB 6981	21.3	—	—
With nylon 6/6	2.0	21.1	14.92
With cell. acetate	4.2	15.4	14.74
With PVC	0.1	12.5	14.06
With PMMA	2.2	10.3	13.86

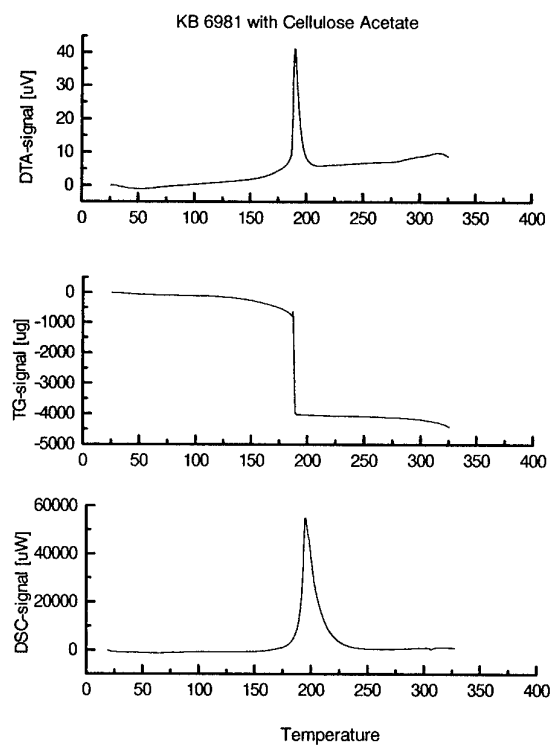
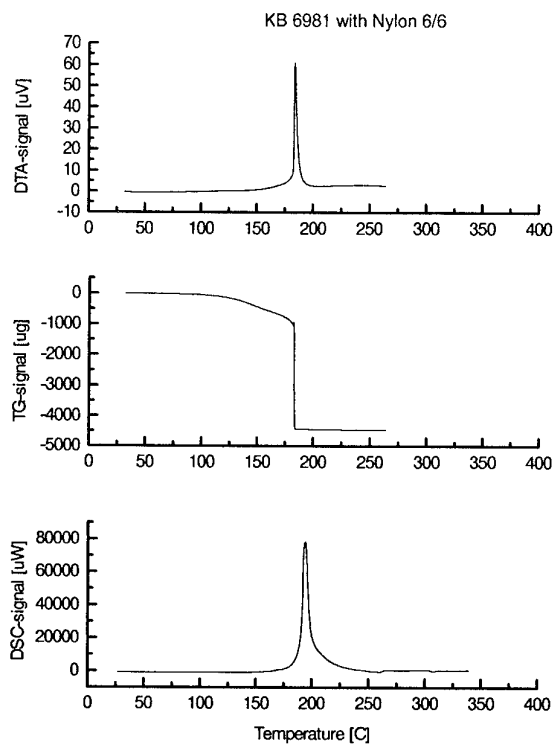
An example of the calculation at 182 °C (arbitrarily choosen) is as follows:

- compound A - KB 6981 (pure) weight loss = 21.3%;
- compound B - PVC (pure) weight loss = 0.1%;
- mixture contains: 66.8% A and 33.2% B;
- calculated weight loss is $(0.213 \cdot 66.8) + (0.001 \cdot 33.2) = 14.06\%$;
- observed weight loss at 182 °C = 12.5%;
- *conclusion*: the mixture of KB6981 with PVC is compatible based on the TG-test.

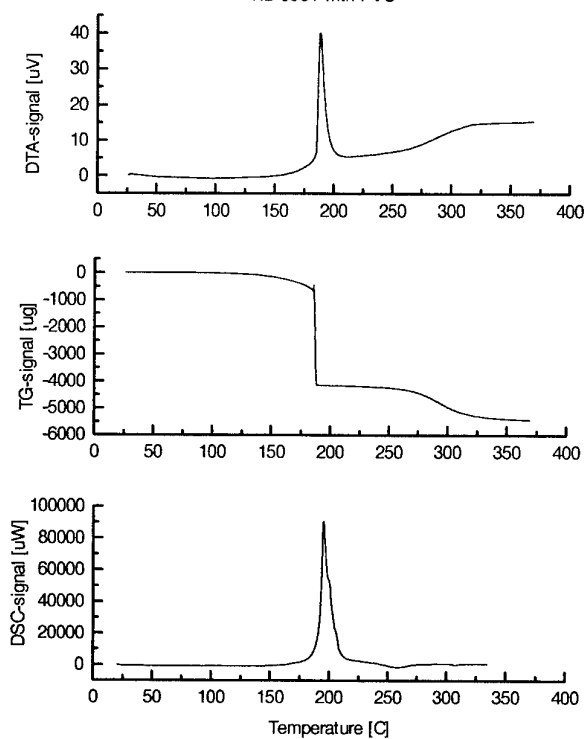
Based on the results of Table 14, it may be concluded that the combinations of KB 6981 with PVC, Cellulose Acetate and PMMA are compatible according to STANAG 4147 (Ref. 4). The mixture with Nylon 6/6 lies in the grey area, therefore kinetic research is advisable. It should be noted that kinetic studies are extremely time-consuming.

An earlier programme (Ref. 6) with KB 6981 also resulted in a compatibility of the propellant with PMMA, PVC and Cellulose Acetate. A relation with Nylon 6/6 is

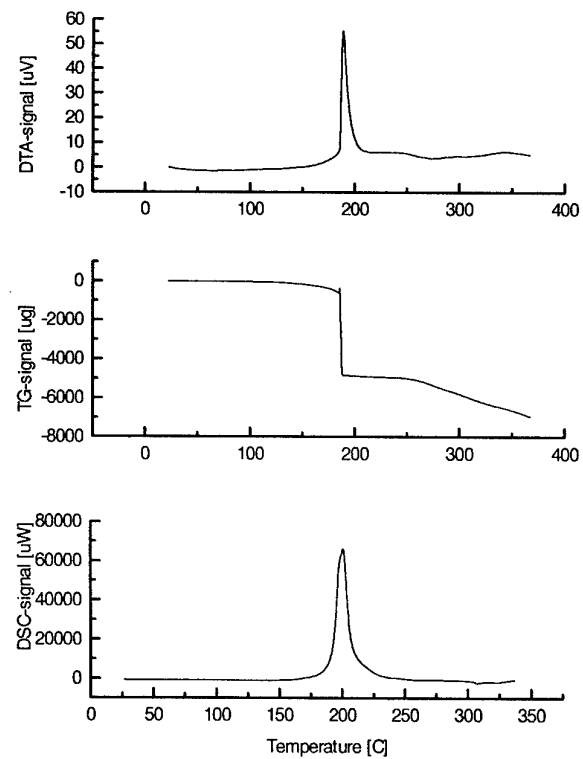
not possible because HFC results (Heat Flow Calorimeter) with that propellant are not available yet.



KB 6981 with PVC



KB 6981 with PMMA



9 Heat capacity measurements

9.1 Theory

One of the basic properties of a material is its heat capacity, C_p . The heat capacity represents the ability of a substance to store heat. It is defined, under constant pressure conditions, as:

$$C_p = dH/dT = dQ/dT$$

where dH/dT represents the change in enthalpy (heat) with respect to temperature. More accurate heat capacity values are becoming increasingly more important as researchers attempt to model (for example Cook-off experiments) the thermal behaviour of materials. A comparison of the experimental heat capacities versus the theoretical heat capacities allows the researcher to learn more about the morphology or structure associated with a substance.

Differential Scanning Calorimetry provides a useful technique for the measurement of heat capacity values based on its ease of use and sensitivity. The current approach for obtaining C_p measurements using DSC entails performing three separate experiments:

- analysis of the baseline of the DSC cell;
- measurement of a heat capacity standard (e.g. sapphire);
- analysis of the sample.

The baseline experiment is required since the characteristics of DSC cells can vary slightly from day-to-day and can significantly affect the experimental C_p -values.

The analysis of a standard, such as sapphire is required in order to provide a greater degree of accuracy. The baseline data is then subtracted from both the sample and standard results and the resulting heat flow values of the sample and standard are then compared, using a simple ratio factor. Based on the well-established heat capacity data for the standard, the heat capacity of the sample can be calculated as a function of temperature.

9.2 Measurement of the heat capacity

The heat capacity of six components is determined by using DSC with aluminium cups.

The investigated samples are the explosives HMX, PBX-RU-113, RDX, TNT and the propellants KB6856 and KB6981.

Table 15: *Cp-values [J/g. °C] of the investigated samples.*

Temp. [°C]	KB 6856	KB 6981	RDX-011	TNT-021	PBX-RU-113	HMX-013
35	1.105	1.296	1.075	1.139	1.212	1.089
40	1.101	1.302	1.046	1.100	1.252	1.076
45	1.112	1.306	1.039	1.088	1.259	1.086
50	1.114	1.310	1.044	1.100	1.265	1.093
55	1.126	1.320	1.048	1.106	1.269	1.113
60	1.134	1.330	1.060	1.123	1.276	1.124
65	1.140	1.335	1.069	1.153	1.287	1.139
70	1.156	1.344	1.085	1.226	1.297	1.158
75	1.170	1.358	1.104	1.300	1.303	1.178
80	1.185	1.371	1.113	2.106	1.321	1.197
85	1.201	1.381	1.128	20.025	1.328	1.214
90	1.211	1.396	1.143	3.464	1.334	1.230
95	1.240	1.419	1.161	1.583	1.354	1.254
100	1.255	1.437	1.178	1.526	1.372	1.275
105	1.269	1.459	1.202	1.537	1.377	1.297
110	1.299	1.492	1.215	1.547	1.405	1.320
115	1.320	1.524	1.232	1.557	1.424	1.342
120	1.333	1.563	1.249	1.565	1.437	1.356
125	1.349	1.603	1.270	1.591	1.447	1.379
130	1.359	1.640	1.295	1.600	1.459	1.392
135	1.377	1.685	1.308	1.620	1.484	1.415

According to Ref. 19, a satisfactory fit function for the Cp values is a third order formula. For the investigated samples, the obtained functions are:

$$\text{KB 6856} : C_p = 1.2001 - 0.0054 * T + 8.839E-05 * T^2 - 2.837E-07 * T^3$$

$$\text{KB 6981} : C_p = 1.2311 + 0.0029 * T - 4.129E-05 * T^2 + 3.341E-07 * T^3$$

$$\text{RDX - 011} : C_p = 1.2325 - 0.0087 * T + 1.193E-04 * T^2 - 3.793E-07 * T^3$$

$$\text{PBX-RU-113} : C_p = 1.1583 + 0.0023 * T - 1.055E-05 * T^2 + 8.377E-08 * T^3$$

$$\text{HMX - 013} : C_p = 1.1499 - 0.0049 * T + 9.252E-05 * T^2 - 3.120E-07 * T^3$$

All the above-mentioned fits are calculated in the temperature range from 35 °C to 135 °C.

TNT differs from the above compounds; the Cp curve could be divided into two parts, namely:

$$\text{TNT-021 (I)} : C_p = 1.4430 - 0.0092 * T - 6.419E-05 * T^2 + 2.155E-06 * T^3$$

$$\text{TNT-021 (II)} : C_p = 1.3415 + 0.0043 * T - 4.621E-05 * T^2 + 2.222E-07 * T^3$$

TNT gives two different curves; the indices are I - before melting and II - after melting.

The curves, with the fit functions, are graphically presented in Figures 6 to 8.

All measurements were performed with a sample mass of about 10 mg. Increasing the sample quantity can advance the measuring precision. On the other hand, if the sample quantity is too small (e.g. primary explosives), the measuring precision will decrease.

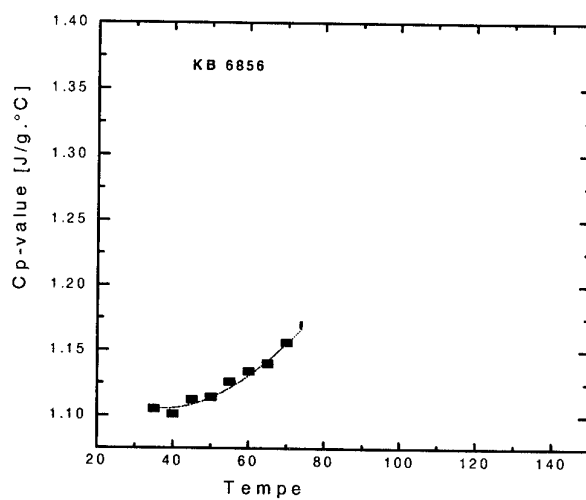


Figure 6: C_p curve (measured and fitted) for propellant KB 6856.

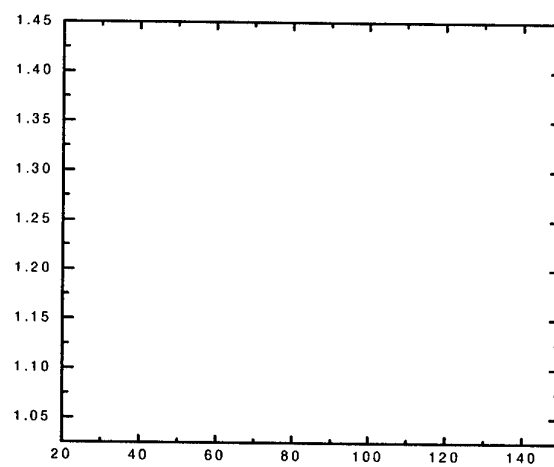


Figure 7: C_p curve (measured and fitted) for explosive HMX.

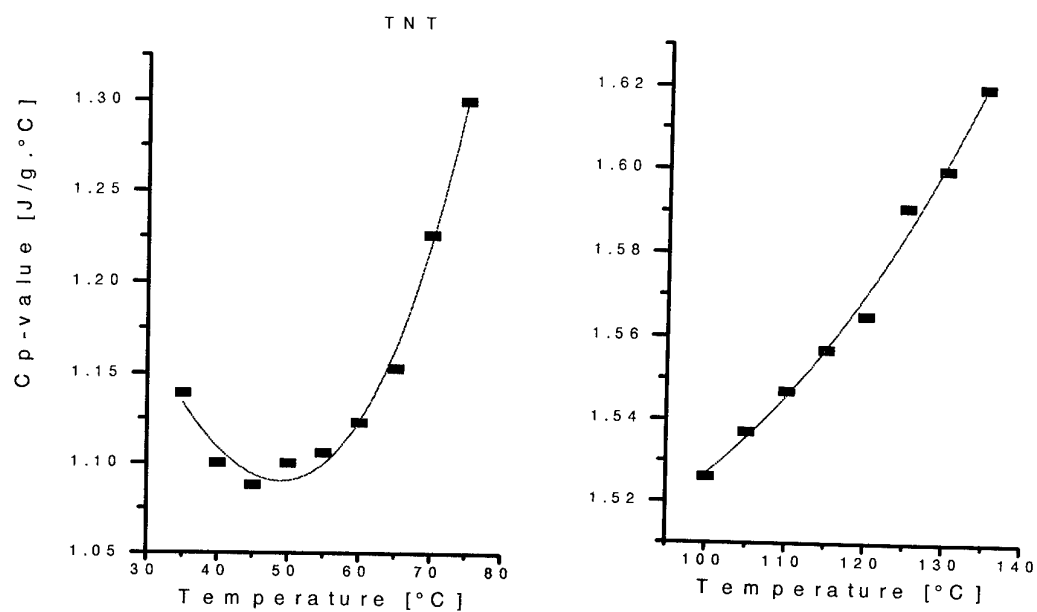


Figure 8: C_p curve (measured and fitted) for explosive TNT.

10 Conclusions/future investigations

According to this programme, the following can be concluded:

- thermal analysis is an excellent technique to obtain data that thermally characterise explosives and propellants;
- testing compatibility with TA can be performed rapidly as a first screening test;
- many problems occur in interpreting the results (e.g. NTO and HMX);
- good measurement of the decomposition enthalpy is only possible by using DSC with closed vessels;
- the DSC measuring of heat capacity has some advantages: the relationship between the heat capacity and temperature can be obtained performing one test, and the quantity of measured sample is also small;
- the DSC method is ideal for determining the heat capacity of explosives and related materials.

Future investigations:

- kinetic analysis of some frequently used explosives, as input for models to describe the experimental results of larger scale tests;
- determination of the auto-ignition temperatures of pyrotechnics;
- extending the database with other explosives and propellants;
- measurement of the exothermic enthalpy using closed vessels, the advantage is that no evaporation can take place;
- to investigate more explosives, to keep the database up-to-date;
- compatibility tests by TA with components in the 'grey area'.

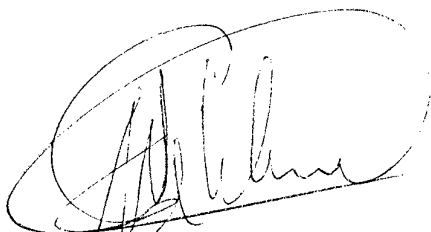
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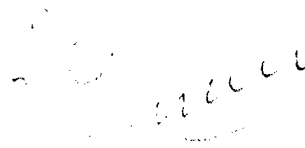
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12 Authentication

This investigation was performed and supervised by W.P.C. de Klerk.
The undersigned herewith declare that the study reported here has been carried out according to the agreed protocol and that this report contains the accurate description of the results.

A handwritten signature in dark ink, appearing to be 'W.P.C. de Klerk', enclosed within a large, loopy oval shape.

W.P.C. de Klerk
Author/Study director

A handwritten signature in dark ink, appearing to be 'Dr. B.J. van der Meer', written in a cursive style.

Dr. B.J. van der Meer
Research coordinator

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